Central Facility for Analytical Microscopy and Microanalysis

Chemical Hygiene Plan
06/14/2011

Updates: _____
_____
_____

1. Please print out and keep this plan (or copies) where everyone working in the lab has access anytime they are working.
2. Have all lab workers review the plan and sign on page iii.
3. Add information and update yearly or more often as necessary.
4. To this document include your lab's Standard Operating Procedures.
5. This is a good place to keep a record of all in-lab training sessions.

Responsibility & Accountability

Revised June 14, 2011 *(must be updated at least annually)*

Departmental & Campus Review

Reviewed by:

Chairperson (or Director) **Harry Green**	**signature**	**date**
K. Bozhilov		6-14-2011
Laboratory Safety Officer (CHO)	**signature**	**date**
Janice T. Leslie	Janice Leslie	6/14/2011
~~**Management Service Officer (or Administrator)**~~ **Janice Leslie** Financial Administrative officer (FAO)	**signature**	**date**
Russell Vernon, Ph.D.		6/21/2011
UCR EH&S Laboratory/Research Safety Specialist	**signature**	**date**

Principal Investigator Review

Reviewed by

		6-14-2011
Krassimir Bozhilov	**signature**	**date**

Chemical Hygiene Plan for the lab of _____CFAMM_____

Laboratory Review

Reviewed by

Lab Staff, Students, Researchers, Visitors & Volunteers engaged in working with laboratory chemicals

Print name	signature	date
Krassimir Bozhilov	[signature]	6-14-2011
Stephen McDaniel	[signature]	6-14-2011
Harry Green		

(Photocopy this page for more signature spaces.)

Central Facility for Analytical Microscopy and Microanalysis
University of California, Riverside
Chemical Hygiene Plan

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1. References

- California Code of Regulations (CCR) Title 8 section 5191, "Occupational Exposure to Hazardous Chemicals in Laboratories" http://www.dir.ca.gov/Title8/5191.html
- California Code of Regulations (CCR) Title 8, "General Industry Safety Orders" http://www.dir.ca.gov/Title8/sub7.html
- University of California, Riverside, Injury and Illness Prevention Program (IIPP) http://www.ehs.ucr.edu/safety/iipp.html
- NFPA 45 - Standard on Fire Protection for Laboratories Using Chemicals, current ed.
- Uniform Fire Code, current ed.

2. Purpose

The purpose of the Chemical Hygiene Plan (CHP) is to minimize exposure of laboratory personnel and students to health and physical hazards presented by hazardous chemicals used in laboratories under the auspices of the CFAMM at the University of California, Riverside, and to comply with the requirements of California Code of Regulations, Title 8 Section 5191.

3. Scope and Application

The CFAMM Chemical Hygiene Plan outlines the minimum health and safety requirements for those laboratories/operations that are engaged in the "laboratory use of hazardous chemicals" under the auspices of the CFAMM at the University of California, Riverside ("the University").

The CHP, which is applicable only to laboratories that meet the definition of CCR Title 8, §5191, is one of the components of the UCR Injury and Illness Prevention Program (IIPP). All departments are required to comply with the IIPP. Laboratory personnel in compliance with the Chemical Hygiene Program are not required to comply with the Hazard Communication component of the IIPP.

Use of sources that produce ionizing or non-ionizing radiation and policies regarding biohazardous materials are addressed in separate documents. These documents, along with this Chemical Hygiene Plan, will be part of the IIPP for each department with laboratories.

The CFAMM has developed and is implementing this Departmental Chemical Hygiene Plan. This plan:

a. Meets the requirements of the UCR CHP; and
b. Designates the following person(s), _Krassi Bozhilov_, responsible for the implementation, oversight, and annual review of the Departmental CHP; and
c. Designates the Principal Investigator (PI), or his/her designee as the person authorized to approve/disapprove laboratory procedures as written in section 12 of this plan, "Circumstances under which prior approval required", and
d. Incorporates standard operating procedures in accordance with section 11 of this plan. It is encouraged, that individual laboratories or small groups of laboratories under a single supervisor develop their own SOPs within their lab specific chemical hygiene plans that are referred to in this document as "individual CHPs".

4. Definitions

Action level - A concentration designated in Title 8, CCR for a specific substance, calculated as an 8-hour time weighted average, which initiates certain required activities such as exposure monitoring and medical surveillance. This is usually half of the value of the Permissible Exposure Limit.

CCR - The California Code of Regulations. Title 8: www.dir.ca.gov/samples/search/query.htm

Department Safety Coordinator (DSC) - Liaison between the individual department and EH&S. The Department Safety Coordinator facilitates the dissemination of safety information/regulations, reporting of hazards, tracking of compliance activities, and coordination of employee training programs within the department.

Designated area - An area that may be used for work with particularly hazardous substances. A designated area may be the entire laboratory, an area of a laboratory, or a device such as a laboratory fume hood.

Hazardous chemical - A chemical for which there is statistically significant evidence, based on at least one study conducted in accordance with established scientific principles that acute or chronic health effects may occur in exposed personnel.

Laboratory - A facility where the "laboratory use of hazardous chemicals" occurs. It is a workplace where relatively small quantities of hazardous chemicals are used on a non-production basis, and can include greenhouses.

Laboratory use of hazardous chemicals - Handling or use of such chemicals in which all of the following conditions are met:

- Chemical manipulations are carried out on a "laboratory scale";
- Multiple chemical procedures or chemicals are used;
- The procedures involved are not part of a production process, nor in any way simulate a production process; and
- Protective laboratory practices and equipment are available and in common use to minimize the potential for employee [and student] exposure to hazardous chemicals.

Laboratory Safety Officer (LSO) - An employee who is designated by the employer, and who is qualified by training or experience, to provide technical guidance in the development and implementation of the provisions of the Chemical Hygiene Plan. Formerly Chemical Hygiene Officer (CHO).

Laboratory scale - Work with substances in which the containers used for reactions, transfers, and other handling of substances are designed to be easily and safely manipulated by one person. "Laboratory scale" excludes those workplaces whose function is to produce commercial quantities of materials.

Laboratory supervisor - An individual that has authority and responsibility for the personnel and procedures conducted in an individual laboratory.

Medical consultation - Consultation which takes place between a licensed physician and an employee [or student] for the purpose of determining what medical examinations or procedures, if any, are appropriate in cases where a significant exposure to a hazardous chemical may have taken place.

MSDS - Material Safety Data Sheet generally available through the EH&S website http://www.ehs.ucr.edu/services/msds.html

Must - Designates a contractual or policy requirement.

Particularly hazardous substances - These include "select carcinogens", "reproductive toxins", and "substances with a high degree of acute toxicity".

Permissible exposure level (PEL) - The maximum permitted 8-hour time-weighted average concentration of an airborne contaminant. Cal/OSHA lists these limits at www.dir.ca.gov/Title8/5155table_ac1.html

Physical hazard - A substance for which there is scientifically valid evidence that it is a combustible liquid, a compressed gas, explosive, flammable, an organic peroxide, an oxidizer, pyrophoric, unstable (reactive), or water-reactive.

Protective laboratory practices and equipment - Those laboratory procedures, practices and equipment accepted by laboratory health and safety experts as effective, or that the employer can show to be effective, in minimizing the potential for employee exposure to hazardous chemicals.

Regulated carcinogen - Any of the following substances or any substance containing any of the following substances: http://www.dir.ca.gov/Title8/sb7g16a110.html

1,2-Dibromo-3-Chloropropane (DBCP); 1,3-Butadiene; 2-Acetylaminofluorene; 3,3'-Dichlorobenzidine (and its salts); 4,4'-Methylene bis(2-chloroaniline); 4-Aminodiphenyl; 4-Dimethylaminoazobenzene; 4-Nitrobiphenyl; Acrylonitrile; alpha-Naphthylamine; Asbestos; Benzene; Benzidine (and its salts); beta-Naphthylamine; beta-Propiolactone; bis-Chloromethyl ether; Cadmium; Coke Oven Emissions; Dibromochloropropane (DBCP); Ethlyene Oxide; Ethylene Dibromide (EDB); Ethylene Oxide; Ethyleneimine; Formaldehyde; Inorganic Arsenic; Methyl Chloromethyl Ether; Methylendianiline; Methylene Chloride; N-Nitrosodimethylamine; Non Asbestiform Tremolite, Anthophyllite and Actinolite; Vinyl Chloride

Regulated Carcinogens used by Central Facility for Analytical Microscopy and Microanalysis

Silicon (IV) oxide

Titanium (IV) oxide

Silicon carbide, beta

DMP-30 (2,4,6-Tri[dimethylaminomethyl]phenol)

Paraformaldehyde, EM Grade, Prill Purified

VCD Vinyl Cyclohexene Dioxide

Beryllium Solid

Reproductive toxins - Chemicals that affect the reproductive capabilities including chromosomal damage (mutations) and effects on fetuses (teratogenesis).

Reproductive Toxins used by CFAMM

Silicon (IV) oxide

Paraformaldehyde, EM Grade, Prill Purified

Other Chemicals Used by CFAMM

Pelco Fast Cure Hardener (Aminoethylpiperazine, nonylphenol)

Pelco Epoxy Resin (Bisphenol A Reaction Product)

Cerium (IV) oxide

Silicon carbide, beta

Palladium

Quartz Sticky Wax

MWS052 Sample Mounting Wax

MWH080 Sample Mounting Wax

MWH135 Wax Quickstick

CS2 Syton

BP08-BP50 Boron Carbide

02-02460-01 Water Soluble Coolant

Dust-Off Plus (Methane, Chlorodifluoro)

Cargille Immersion Oil Type NVH

Gold

Kodak Photo-Flo 200 Solution

Kodak Fixer

Pelco Colloidal Graphite

Indicating Silica Gel

Pelco Colloidal Silver

Kodak D-19 Developer

Araldite 502

D.E.R. 736 Epoxy Resin

DMAE, Dimethylaminoethanol

Eponate 12 / Medcast

FORMVAR 15/95E Resin

Ethyl Alcohol, Anhydrous, Denatured

Glutaraldehyde

Lowicryl K4M Kit

Nadic Methyl Anhydride

Nonenyl Succinic Anhudride Modified

Osmium Tetroxide

Propylene Oxide

Ruthenium Red

Sodium Phosphate, Monobasic, Anhydrous

Sodium Cacodylate Trihydrate

Sodium Hydroxide	Uranyl Acetate
Sodium Sulfite	M-Bond 610 Adhesive
Toluidine Blue	Platinum

Select carcinogen - Any substance that meets one of the following criteria:

- It is regulated by Cal/OSHA as a carcinogen (see regulated carcinogens listed above); or
- It is listed under the category "known to be carcinogens", in Annual Report on Carcinogens by the National Toxicology Program (NTP) (latest ed., http://ntp.niehs.nih.gov/?objectid=72016262-BDB7-CEBA-FA60E922B18C2540); or
- It is listed under Group 1 ("carcinogenic to humans") by the International Agency for Research on Cancer Monographs (IARC) (Vol. 1-48 and Supplements 1-8); or
- It is listed in either Group 2A or 2B by IARC (http://monographs.iarc.fr/) or under the category, "reasonably anticipated to be carcinogens" by NTP (http://ntp.niehs.nih.gov/?objectid=72016262-BDB7-CEBA-FA60E922B18C2540) and causes statistically significant tumor incidence in experimental animals in accordance with any of the following criteria:
    a. After inhalation exposure of 6-7 hours per day, 5 days per week, for a significant portion of a lifetime to dosages of less than 10 mg/m^3;
    b. After repeated skin application of less than 300 mg/kg of body weight per week; or
    c. After oral dosages of less than 50 mg/kg of body weight per day

Shall - Designates a regulatory mandate.

Should - Designates a recommendation contained in the regulations or a recommendation from a recognized industry standard.

Substances which have a high degree of acute toxicity - Substances that are "Highly Toxic" under the definitions in Appendix A of CCR Title 8 section 5194 (http://www.dir.ca.gov/Title8/5194a.html).

Will - Designates a UCR policy or standard practice.

5. Responsibilities

The **Chancellor** is responsible for the implementation of the University's Environmental Health and Safety policies at all facilities under campus control.

The **Vice-Chancellors, Deans, and Executive Officers** are responsible for insuring that departments/units under their authority that are engaged in the laboratory use of hazardous chemicals comply with the Chemical Hygiene Plan (CHP) applicable to each unit.

Department chairpersons, department heads, in areas engaged in the laboratory use of hazardous chemicals are responsible for assigning a person to be the Departmental Laboratory Safety Officer and meeting with that person at least semi-annually to discuss laboratory safety.

Department chairpersons, department heads, unit heads, managers, principal investigators, and laboratory supervisors in areas engaged in the laboratory use of hazardous chemicals are responsible for establishing, implementing and maintaining a CHP and ensuring compliance with that plan.

Employees and students engaged in the laboratory use of hazardous chemicals are responsible for complying with departmental CHPS and individual SOPs.

The **Departmental Laboratory Safety Officer** is responsible for preparation and annual review of the departmental Chemical Hygiene Plan and for providing technical assistance to laboratory workers. The Laboratory Safety Officer for the CFAMM is Krassi Bozhilov.

The **Environmental Health and Safety Office (EH&S)** is responsible for updating the campus CHP and assisting in the development and annual review of departmental and individual Chemical Hygiene Plans. EH&S has assigned the Laboratory / Research Safety Specialist to act as the liaison and coordinator to the departmental Laboratory Safety Officer and as the chair of the Laboratory Safety Officer Committee. The UCR, EH&S Laboratory / Research Safety Specialist provides technical guidance in the development and implementation of this Chemical Hygiene Plan. EH&S is also responsible for providing Material Safety Data Sheets and other hazard information on request (http://www.ehs.ucr.edu/services/msds.html), monitoring and inspecting to determine compliance with federal, state, and local health and safety regulations (http://www.ehs.ucr.edu/safety); and assisting with the development of safety training and education programs for CHP participants.

6. Information and Training

The CFAMM shall provide to all persons under the auspices of the department involved in the laboratory use of hazardous chemicals, information and training to apprise these persons of the hazards of the chemicals in their work area. This training is a subset of the general safe work practices training required by the Injury and Illness Prevention Program and the Integrated Safety and Environmental Management System. Laboratory personnel who have had training under the Chemical Hygiene Plan are not required to have Hazard Communication training as long as all their work with hazardous materials falls within the scope and definition of "laboratory use".

The information and training shall be provided to the laboratory personnel or student at the time of initial assignment to the laboratory, prior to assignments involving new exposure situations, and upon changes in the procedures used and/or new hazards present in the laboratory. Annual refresher training on the CHP is required for all laboratory employees and students.

Information provided shall include:

a. The physical location and availability of "Occupational Exposure to Hazardous Chemicals in Laboratories" regulation, CCR Title 8, Article 110, Section 5191. www.dir.ca.gov/Title8/5191.html
b. The location and availability of the Chemical Hygiene Plan. The CFAMM Chemical Hygiene Plan is located in the CFAMM lab, room B116 Bours Hall, and is available from 8:00 a.m. to 5: p.m. Monday-Friday.
c. The permissible exposure limits and recommended exposure limits for chemicals www.dir.ca.gov/Title8/5155.html and www.dir.ca.gov/Title8/5155table_ac1.html
d. Signs and symptoms associated with exposures to hazardous chemicals used in the laboratory; as found in the "Health Effects" section of the Material Safety Data Sheet for each chemical.
e. The physical location and availability of known reference material(s) on the hazards, safe handling, storage and disposal of hazardous chemicals found in the laboratory including, but not limited to, Material Safety Data Sheets (MSDSs) received from the chemical supplier. Each laboratory shall have MSDSs for chemicals used in that laboratory readily available in a clearly marked notebook or file in the laboratory or online. Additional MSDSs are available at the EH&S office and online through the EH&S website http://www.ehs.ucr.edu/services/msds.html. Additional safety references are available in the Science Library and are listed in Appendix B.

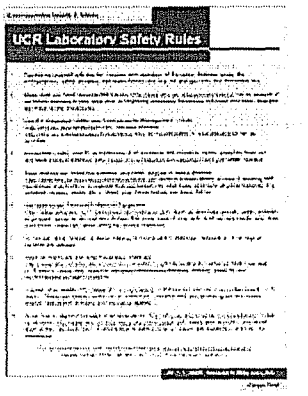
Training shall include:

a. Methods and observations that may be used to detect the presence or release of a hazardous chemical;
b. The physical and health hazards of chemicals in the work area;
c. The measures that can be taken to protect oneself from these hazards, including specific procedures the University has implemented to protect persons from exposure to hazardous chemicals, such as engineering controls, appropriate safe work/lab practices, emergency procedures, and personal protective equipment to be used;
d. The applicable details of the Chemical Hygiene Plan (in particular relevant SOPs)

Training shall be documented in accordance with the Injury and Illness Prevention Program. The CFAMM office maintains records of EH&S safety training completed by faculty, staff and students and should be notified when such training is completed. Each principle investigator responsible for laboratory space must keep documentation of training in their own laboratories that includes the name of the trainee, the date, and the procedures covered.

7. Basic or General Laboratory Safety Rules

www.ehs.ucr.edu/laboratory/laboratorysafetyrules.pdf



- Know the location of emergency showers, eyewashes, first aid kits, emergency exits, spill kits, telephone and fire alarm pull stations. Know the location of the nearest and the next-nearest telephone, eyewash, and emergency shower. In an emergency, dial 911 from a campus phone. (From a cell phone call 951-827-5222)
- Know the location of Material Safety Data Sheets http://www.ehs.ucr.edu/services/msds.html. Ensure that you have been trained on this Chemical Hygiene Plan.
- No food, beverage, smokeless tobacco, or application of cosmetics is allowed in the laboratory. No smoking is allowed in any area of a laboratory. Always wash hands after working with chemicals, even when gloves have been used.
- Dress appropriately. Long hair, neckties, or loose clothing should be tied back or otherwise secured. No sandals, open-toed or perforated shoes are allowed to be worn in the laboratory. Lab coats will be worn over short skirts (above the knee), shorts, or exposed midriffs.
- Appropriate eye protection (safety glasses at a minimum) will be worn at all times in laboratories; see Section 16 below and any applicable SOPs.
- Wear the appropriate personal protective equipment for the chemicals you are working with. When wearing contact lenses, be sure to wear safety goggle to protect your sight.
- Comply with warning signs and labels.
- Laboratories with special or unusual hazards shall be posted with appropriate warning signs including but not limited to: laser warning signs, radioactivity warning signs, biological hazard warning signs, etc. The EH&S Hazardous Materials Program (http://www.ehs.ucr.edu/hazardousmaterials) has a Placard Program to assist in the compliance with this requirement (http://econtact.ucr.edu/).
- Do not directly smell or taste any chemical.
- Do not pipette or siphon by mouth.
- Containers shall be kept closed when not in active use.
- When diluting strong acids, it is generally safer to add acid to water as water has a higher heat capacity and can absorb the heat generated by the dilution reaction.
- Perform only those experiments or procedures you are authorized to do by the person in charge of the lab.
- Report all injuries, fires, and accidents to your supervisor or instructor immediately. All fires use of fire extinguisher and must be reported to the Campus Fire Marshal at 951-827-6309 as soon as possible.
- If you have a question about a procedure or the hazards of a chemical, ask your supervisor or instructor before performing the procedure.

8. Labeling

All containers of hazardous substances shall be appropriately labeled. The manufacturer's label shall not be removed from a container as long as the material or residues of the material remain in the container. The following information is to be provided on the manufacturer's label:

1. The identity of the hazardous substance.
2. Appropriate warning words and statements.
3. Appropriate precautionary measures.
4. Name and address of manufacturer or importer.

The receiver will write the date received, and opened on the label of chemicals that expire or can convert to a reactive material upon standing, such as diethyl ether, tetrahydrofuran, 1,4-dioxane, diisopropyl ether and other peroxide-forming substances. See Appendix A-III.

All containers into which hazardous substances are transferred or prepared shall be labeled with the following information:

a. The identity and concentration of the hazardous substance.
b. A description of the hazards and precautionary measures. In containers smaller than one quart, descriptions can be limited to signal words such as "FLAMMABLE, CORROSIVE, TOXIC"; consult a recent original container, the Aldrich Chemical catalog, the MSDS, or contact EH&S (951)827-5528 www.ehs.ucr.edu .
c. It is good practice to put the date received, prepared or transferred on the label. This is required for peroxidizable chemicals such as ethers; see Appendix A-III below.
d. It is recommended that the responsible person's name, building, room number, and telephone number be on the container label.

All containers in which hazardous waste is collected shall be labeled with a completed hazardous waste tag. The campus uses an On-line Hazardous Waste Tag Program (http://otp.ucop.edu) to track waste and avoid storage and labeling violations. Guidance on how to sign up for the OTP and use it is available both through an on-line manual (http://www.ehs.ucr.edu/forms/otpmanual.pdf) and an on-line tutorial (http://www.ehs.ucr.edu/training/online/otp).

Labeling is not required for portable containers into which hazardous chemicals are transferred from labeled containers, and which are intended only for the immediate use of the individual who performs the transfer.

Where hazardous materials are transported by piping systems, above ground pipes shall be labeled:
a. Preferably by stenciling the name of the chemical and an arrow indicating direction of flow of the material.
b. Alternatively, by permanently marking or attaching tags of metal or other such suitable material naming the material transported on or near valves.

9. Chemical Procurement and Distribution

Each individual Chemical Hygiene Plan should include protocols used by the laboratory or laboratories under the Plan for procuring and distributing hazardous chemicals. The Chemical Procurement and Distribution protocol should include the following considerations:

a. Substitute with less hazardous chemicals where possible.
b. Limit purchases to amounts that can forseeably be used in the next year or less.
c. Utilize microscale techniques where feasible.
d. Ensure that facilities and equipment are adequate for the intended use of the chemical.
e. Ensure that equipment such as bottle carriers, carts with spill containment, or specialized containers are available for use if needed.
f. Attempt to obtain an agreement in writing from the donor of free hazardous materials to accept the return of any unused portions.
g. Ensure that a current MSDS is available or requested from the manufacturer for each chemical purchased. MSDSs are available through http://www.ehs.ucr.edu/services/msds.html.

Surplus Chemicals
The EH&S department maintains a chemical reuse program. When disposing of reagent quality chemicals, identify them to the waste program as having the potential for re-use. Reassigned, relocated, or departing researchers are responsible for the proper disposition of their chemical and hazardous waste stocks prior to departure. The departmental Laboratory Safety Officer shall be responsible for the evaluation, redistribution, and/or disposal of other abandoned chemicals discovered within departmental laboratories according to procedures outlined in the Chemical Hygiene Plan.

Hazardous Materials Inventory
California Law requires an inventory of hazardous materials. Each lab within the department is encouraged to complete an inventory online at: http://ucriverside.ecompliance.net If you need assistance or this is the first time the lab is completing an inventory, obtain a username and password from the Hazardous Materials Specialist at 827-5528. This inventory should be updated and kept current by making changes online through the EH&S website . Explanations of materials to be reported are also available on the web site.

10. Storage of Chemicals in Laboratories

General Guidelines for Storage of Chemicals in Laboratories

a. Shelves used for chemical storage shall be of substantial construction and adequately braced. Shelves shall be provided with a lip or guard when used for the storage of individual containers, except when containers are stored in a hazardous materials storage cabinet or other furniture designed to prevent containers from falling off the shelf during seismic activity.
b. Containers are to be arranged in such a manner as to allow all labels to be seen. When this is impossible, the shelf may be labeled with a list of contents.
c. Chemicals should not be stored on the floor and should not protrude into traffic areas.
d. Materials sensitive to light shall be stored in containers designed to protect the contents from light.
e. Food shall not be stored or consumed in areas where toxic chemicals are used or stored. (http://www.dir.ca.gov/title8/3368.html) Avoid eating, drinking, gum chewing, or application of cosmetics in areas where laboratory chemicals are present; wash hands before conducting these activities. Avoid storage, handling, or consumption of food or beverages in storage areas, refrigerators, glassware or utensils that are also used for laboratory operations. (http://www.dir.ca.gov/title8/5191a.html). Hallways and stairways should not be used as storage areas.

Storage of flammable liquids in laboratories

a. Storage of more than 10 gallons of flammable and combustible liquids (with a flash point less than 100° F) and 120 gallons of combustible liquids (with a flash point of less than 140 degrees F) shall be in a flammable liquid storage cabinet meeting the requirements of CCR Title 8, Section 5533. The manufacturer usually specifies that the cabinets meet the requirements of OSHA or NFPA 30 -Flammable Liquids Code.
b. Flammables shall not be stored in the same cabinet as oxidizers, strong acids or water-reactive materials in a manner in which they could mix during a catastrophic event as an earthquake.

Storage of corrosives in laboratories

a. Corrosives ought not be stored under sinks or in other areas where plumbing, equipment, or shelving could be damaged by corrosive effects. Containers of hydrochloric acid, fuming acids, or other volatile corrosives should be securely capped.
b. Strong acids (pH <2) and strong bases (pH >12.5) shall be stored separately in a manner precluding their mixing during a catastrophic event as an earthquake. Where amounts are small, separation can be achieved by either all the acids or all the bases being stored in separate containment such as bottle carriers, spill pans, or other secondary containment. Organic acids (e.g., acetic) should be stored with organics, not with strong inorganic acids.

11. Standard Operating Procedures

Each Individual Chemical Hygiene Plan shall have standard operating procedures (SOPs) that are relevant to safety and health considerations of the laboratory or laboratories covered by the specific plan and capable of preventing health hazards from exposure. These SOPs should address work practices and policies necessary to protect personnel and students from the exposure to hazardous materials in the laboratory. Sample SOPs that may be adopted or adapted for use in individual plans are available in Appendix A of this plan. Additional specific SOPs, which may be necessary to protect personnel and students in the laboratory must be developed for individual CHPs. Environmental Health & Safety can assist in locating informational resources and developing new and specific SOPs. Any SOPs that are developed should follow the Integrated Safety & Environmental Management System process and be shared with EH&S. **See Appendix A, Section VI for the CFAMM's Standard Operating Procedures.**

12. Circumstances Under Which Prior Approval is Required

Each departmental or individual Chemical Hygiene Plan shall include designated persons who have authority under the plan to approve laboratory procedures and allow continuance of laboratory procedures under the

conditions specified in this section. There may be as many designees as needed by the department. The Environmental Health and Safety Office will not have final approval for research projects or laboratory procedures. EH&S is available on request to provide information or recommendations to assist designated persons in making approval decisions.

Prior approval will be obtained from the appropriate designee for the following:

1. For new laboratory procedures.
2. When it is likely that the "Action Level" or "Permissible Exposure Limit" for a chemical may be exceeded.
3. For changes in procedures that may affect reaction rates, changes in temperatures, or flammability.
4. When ingredient chemicals in a procedure are substituted, added or deleted, or the amounts of chemicals are significantly increased or decreased.
5. For operations that are to be left unattended
6. For operations to be performed by an individual working alone in a laboratory.
7. For operations involving work with "particularly hazardous substances" as outlined in section 14 of this document.

Approval to continue or proceed will be obtained from the appropriate designee:

1. When there is a failure of equipment, especially safety control measures such as fume hoods, clamp apparatus, or temperature control.
2. When the procedure produces unexpected results
3. When signs and symptoms of chemical exposure are experienced.

13. Criteria to Determine and Implement Control Measures

Hazard determination

1. Labels containing substance identification and hazard information on incoming material shall not be removed or defaced as long as any material or residue remains in the container.
2. All Material Safety Data Sheets (MSDS's) received shall be retained. MSDS's are available on-line through http://www.ehs.ucr.edu/services/msds.html and www.ucmsds.com. Receivers of MSDS's that are not available through these websites shall retain a copy of the MSDS and send a clean copy to the Hazardous Materials Manager (http://www.ehs.ucr.edu/hazardousmateraisl) at Environmental Health and Safety. If hazardous chemicals are going to be used where Internet access is not readily available, such as at a field station, copies of the MSDS's shall be printed and taken with the workers.
3. Exposure levels and effects on humans will be determined using information sources suggested in Appendix B of this document and in accordance with Appendices A & B to CCR Title 8 section 5194 (Appendix C of this document). Permissible exposure limits are available on the California Occupational Safety and Health Administration web site at http://www.dir.ca.gov/Title8/5155table_ac1.html.
4. Physical hazards presented by chemicals will be determined using information sources suggested in Appendix B of this document.

5. Implementation of control measures
6. Sufficient general ventilation, local ventilation, isolation, or respiratory protection must be used to protect against airborne contaminants.
7. Isolation, shields or barriers, and chemical protective clothing and equipment may be selected for use with chemicals that may be absorbed in injurious levels by the skin. Permeation and degradation factors of protective clothing and equipment will be considered when making selections.
8. Control measures for fire and explosion hazards may include ventilation, controls as recommended by NFPA 70 - National Electrical Code, NFPA 45 – Fire Protection for Laboratories Using Chemicals, and other recognized standards and recommendations as warranted.

14. Provisions for Protection From Particularly Hazardous Substances

This section will apply to laboratory use of chemicals defined as "particularly hazardous substances". These include "select carcinogens", "reproductive toxins", and "substances with a high degree of acute toxicity".

Establishment of a designated area

1. A designated area may be the entire laboratory, an area of the laboratory, or a fume hood or glove box,
2. Storage or consumption of food, storage or use of containers of beverages, storage or application of cosmetics, smoking, storage of smoking materials, tobacco products or other products for chewing, or the chewing of such products, shall be prohibited in designated areas.
3. Use of particularly hazardous substances shall be limited to designated areas.
4. The location of the designated area shall be posted and persons working within the designated area shall be informed of the hazardous substances used there. A sample posting for designated areas is provided in Appendix D.

Use of containment devices and methods

1. Containment devices and methods should be used to minimize exposure to persons and to prevent contamination of areas outside the designated area.
2. Use of particularly hazardous substances in a manner which may produce vapors, dusts, mists, particularly hazardous gases or other airborne particulates shall be under a laboratory hood, in a glove box, or in a closed system.
3. Laboratory fume hoods in which other substances are used shall have an average face velocity of 100 linear feet per second, with no area falling below 70 linear feet per second.
4. Mechanical pipetting aids shall be used for all pipetting procedures (no mouth pipetting).
5. Work surfaces that may become contaminated with particularly hazardous substances shall be protected from contamination.
6. When working with regulated carcinogens, laboratory vacuum systems shall be protected with a double cold trap or with disposable absolute filters. When working with beta- propiolactone, bis-chloromethyl ether, methyl chloromethyl ether, or ethyleneimine, a double cold trap shall be used.
7. Persons working in the designated area shall remove protective equipment and wash their hands and forearms before engaging in other activities or before eating, drinking, smoking, or using toilet facilities.

Procedures for safe removal of contaminated waste

1. All waste contaminated with particularly hazardous substances should be collected in impervious containers that are compatible with the contaminant.
2. Wastes contaminated with different substances that are incompatible shall not be placed in the same container.
3. Contaminated sharps, contaminated broken glassware, etc. should be placed in a sturdy container such as a cardboard box and sealed before placing in a waste collection container. Puncture-proof containers for hazardous hypodermic needles can be obtained from EH&S.
4. All containers should be labeled with the contaminant(s), and if the contaminant is a carcinogen, the words "Cancer-suspect Agent".
5. Hazardous waste shall not be accumulated at satellite locations (e.g., laboratories) for longer than 6 months and acutely hazardous waste volumes cannot exceed 1 quart. Waste will be collected when the box associated with the accumulated container is selected and the "Checked items ready for disposal" button is clicked in the OTP system (http://otp.ucop.edu).

Decontamination procedures

1. Laboratory workers should consider whether or not decontamination of designated areas is appropriate. Decontamination procedures and use of PPE will vary according to the type of substance used (consult with EH&S).
2. Small spills should be cleaned up immediately and the area decontaminated.
3. Large or particularly hazardous substance spills which require the activation of the UCR Emergency Response Plan (http://www.ehs.ucr.edu/resources/emergencyprocedures.pdf) will be cleaned up in accordance with that plan.

15. Housekeeping, Maintenance, Inspections, and Monitoring

Housekeeping

1. Laboratories should be kept free of clutter. Working areas should be cleaned up at the end of each operation and at the end of each day.
2. Small spills of chemicals should be cleaned up immediately. Spills which require measures beyond general housekeeping by laboratory personnel to clean up should be reported to EH&S 827-5528 (after hours 827-5222) or, in case of emergency, call 9-911 from a campus phone.
3. Safety showers, eyewashes, and fire extinguishers shall be free from any obstruction that would prevent access and use. Access to emergency exits shall be kept clear at all times.
4. Circuit breaker panels shall have an unobstructed clearance of 30".
5. The floor shall be kept clean and free of slip hazards by reasonable cleaning and immediate clean up of spills.
6. Old containers, compromised containers, and chemical wastes should be disposed of promptly and not be allowed to accumulate. Hazardous waste materials should be disposed of in accordance with section 19 of this plan.

Inspection and Maintenance of Protective Devices

1. Temperature control and over temperature shutoff devices on heating equipment should be tested in accordance with manufacturer recommendations to ensure proper operation.
2. All automatic shutoff devices should be tested in accordance with manufacturer recommendations to ensure proper operation.
3. Records of inspection and testing of above equipment will be maintained Bourns B116, for 3 years.
4. Fume hood performance will be evaluated by EH&S. (www.ehs.ucr.edu/safety/Fume_Hoods/fumehood.html)
5. Explosion shields and isolation devices should be visually inspected by the user for cracks or other damage before each use.
6. Safety showers and emergency eyewashes shall be inspected monthly by Physical Plant personnel.
7. (www.pplant.ucr.edu/facilities/plumbing.htm). Tags indicating date inspected and the inspector will be attached on or near the shower or eyewash.

Laboratories shall be inspected at least annually by the department. A checklist to assist in inspections is provided in Appendix E.

Problems with utilities such as water, air, building vacuum, etc? Contact Physical Plant at (951) 827-4214 www.pplant.ucr.edu

16. Personal Protective Equipment (PPE)

Respiratory protective equipment - The Office of Environmental Health and Safety will act as the sole source for purchasing, fitting and approving the use of all respiratory equipment and for training University personnel and students in its proper use and maintenance. (As required by UCR Policy and state law http://www.ehs.ucr.edu/safety/programs.html)

1. Eye and Face Protection Eye and face protection shall be required where there is a reasonable probability of injury that can be prevented by such equipment. Appropriate eye protection shall be worn at all times in laboratories. At a minimum, safety glasses with side shields shall be worn.
2. "Design, construction, testing and use of devices for eye and face protection shall be in accordance with American National Standard for Occupational and Educational Eye and Face Protection, (ANSI) Z87.1" Compliant eye or face protection is usually marked with "Z87".
3. Selection of the proper type of eye and face protection should be in accordance with ANSI Z87.1 that is available on campus at EH&S or in the Science Library.

4. Skin protection

5. When working with those substances that are readily absorbed by the skin, or with substances that are highly corrosive to the skin, appropriate protection shall be provided to protect the laboratory worker from skin contact.
6. Skin protection may take the form of shields, isolation of the procedure, gloves, aprons, or other such protective equipment.
7. Gloves and aprons should be selected according to the chemical resistance of the protective material to the chemical to be used. This information can be obtained by the supplier of the product and is usually presented in a chemical resistance chart in the supplier's catalog. The Environmental Health and Safety Office can assist in the selection of proper personal protective equipment.

17. Medical Consultation and Examinations

The University shall provide all persons involved in the laboratory use of chemicals an opportunity to receive medical attention, including any follow-up examinations that the examining physician determines to be necessary, under the following circumstances:

1. Whenever a laboratory employee or laboratory student develops signs or symptoms associated with a hazardous chemical to which the employee or student may have been exposed in the laboratory;
2. Where industrial hygiene monitoring in a laboratory reveals an exposure level routinely above the action level (or in the absence of an action level, the Permissible Exposure Limit http://www.dir.ca.gov/Title8/5155table_ac1.html); or
3. Whenever an event takes place in the laboratory such as a spill, leak, explosion or other occurrence resulting in the likelihood of a hazardous exposure.

All medical examinations in accordance with section 18 of this document shall be performed by or under the direct supervision of a licensed physician and shall be provided at no cost to the exposed laboratory employee or laboratory student.

The University shall provide the following to the physician:

1. The identity of the hazardous chemical(s) to which the laboratory employee or laboratory student may have been exposed and a copy of the MSDS, if available;
2. A description of the conditions under which the exposure occurred including quantitative exposure data, if available; and
3. A description of the signs and symptoms of the exposure that the laboratory employee or laboratory student is experiencing, if any.

Exposure records shall be maintained by Environmental Health & Safety (http://www.ehs.ucr.edu/biosafety) and medical consultation records shall be maintained by the physician conducting any examination in accordance with CCR Title 8, section 3204. (http://www.dir.ca.gov/Title8/3204.html) Information about this process can be obtained from EH&S.

18. Spills and Accidents

Ensure that you know the details of the departmental Emergency Action/Fire Prevention Plan applicable to your location. The campus guidance is on-line at: http://www.ehs.ucr.edu/resources/emergencyprocedures.pdf. Know at least two evacuation routes from your location. Know the location of emergency showers, eyewashes, first aid kits, emergency exits, spill kits, and fire alarm pull stations. Know the location of the nearest and the next-nearest telephone, eyewash, and emergency shower. In an emergency, dial 9-911 from a campus phone.

You are expected to be familiar with the hazards of the materials in your labs.

1. Consult Materials Safety Data sheets (http://www.ehs.ucr.edu/services/msds.html) to obtain this information.
2. Select the appropriate response actions based upon the hazards of the materials involved.
3. Special hazards, such as hydrofluoric acid, require specific preparation and first-aid supplies. Discuss these with your Laboratory Safety Officer (http://www.ehs.ucr.edu/services/lso.html).

Fires and Chemical Spills

1) Alert personnel in the immediate vicinity
2) Confine the fire or chemical spill, if possible to do so without endangering personnel.
   a) Keep yourself between the emergency and an exit while attempting to confine the emergency to avoid being trapped.
   b) If you have been trained to put out small fires or use an extinguisher, fight the fire if you are confident that you will be able to put it out.
   c) If emergency is inside a hood, close the sash, if possible.
   d) Close lab doors, if possible, to prevent spread of smoke or vapors into adjoining rooms and corridors.
   e) For flammable liquid spills, shut off ignition sources, if possible. Avoid unplugging equipment due to possible electrical arc between receptacle and plug. Turning off breaker will work.
3) Evacuate the emergency area. If in doubt, evacuate the building. To evacuate a building, pull the nearest fire alarm pull station on your way out.
4) Summon aid.
   a) For emergencies that require response from the fire department, police department, or paramedics, dial 9-911 from a campus phone.
5) For other emergencies or incidents, call EH&S at 827-5528, or campus police 827-5222 after hours.

Clothing Fires

1. Stop the person on fire from running; even from running to a fire blanket.
2. Drop the person to the floor or other horizontal surface to prevent flames and hot gases from rising to the face and head.
3. Roll the person to snuff out the flames. Blankets can be effective if brought to the person. Safety showers may be effective if within a travel time of one or two seconds.
4. Cool the person by removing smoldering clothing that has not adhered to the skin, and by using water and ice packs.
5. Get medical assistance. Call 911 from a cell phone or 9-911 from a campus phone.

Chemical splash

1. Chemical splashes in the eyes
   - Immediately wash the eyes with potable water for at least 15 minutes.
   - Forcibly hold the eyelids open and tell the injured person to roll his/her eyes while continuously irrigating.
   - Do not use any substance other than potable water to wash the eyes.
   - Get medical assistance.
2. Chemical splashes on the skin
   - Remove chemical contact with the skin by brushing off dry and water reactive chemicals and removing contaminated clothing and protective equipment that can be removed quickly (1 second or less).
   - Flush the splashed area with large amounts of potable water. Never use anything other than water or mild soap and water to clean chemicals from exposed skin.
   - Remove protective eyewear under the emergency shower as quickly as possible when chemicals have entered the eyes. In cases where the eyewear has not been breached by the chemical, remove the protective eyewear after head and face have been thoroughly washed.
   - Wash with potable water for 15 minutes or longer. Wash any part of the skin that may have had chemical contact or contact with contaminated wash water. Remove any clothing that may have come in contact with the chemical or contaminated wash water under the emergency shower.
   - Washing should give special attention to areas that may by missed such as underneath the earlobes, underneath the arms, the crotch, between the toes, the creases at the sides of the nose, a deep cleft in the chin, etc.
   - Get medical assistance. Provide MSDSs for the involved chemicals to medical personnel.
   - If the emergency water used for flushing is cold, the injured person should be treated for shock on completion of washing.
   - If a splash causes a thermal burn as well chemical burn, be sure to advise the attending medical personnel the nature of the chemical exposure.
   - After washing of the victim is completed, rescuers need to wash themselves to prevent injury from diluted chemical washed off from the victim.

Work-related injury or illness

- For dire emergencies call 911 from a campus phone (or 951-827-5222 from a cell phone)and request an ambulance.

Medical Treatment Facilities for Employees

Parkview Occupational Medicine
9041 Magnolia Avenue, Suite 107
Riverside, CA 92503
Phone: (951) 353-1021
Hours: Weekdays: 8 a.m. to 9 p.m.; Weekends: 9 a.m. to 6 p.m.
After hours call (951) 351-7726

Central Occupational Medicine Providers (COMP)
4300 Central Avenue
Riverside, CA 92506
Phone: (951)222-2206
Hours: 24 hours a day, 7 days a week.
Transportation can be requested by calling this facility in advance.

IN CASE OF EMERGENCY OR LIFE-THREATENING INJURIES:

Riverside Community Hospital
4445 Magnolia Avenue
Riverside, CA 92507
Phone: (951)788-3000
Hours: 24-Hour Emergency Care
(Follow up treatment should be obtained at Central Occupational Medicine Providers or Parkview Occupational Medicine)

19. Hazardous Waste Disposal

Hazardous Waste Disposal guidance is available on the Environmental Heath and Safety website: www.ehs.ucr.edu. A good place to start is the Waste Management program and the summary of disposal requirements for UCR laboratories: www.ehs.ucr.edu/waste and www.ehs.ucr.edu/services/waste.html

Liquid Chemical Hazardous Waste Collection

1. Hazardous wastes shall be collected in containers that are compatible with the intended contents and that are in good condition.
2. Materials placed in the same collection container shall be compatible with all other materials in the container.
3. Containers shall be labeled with the On-line Hazardous Waste Program at http://otp.ucop.edu.
4. All chemical reactions should be complete prior to introduction into collection containers.
5. Whenever possible, markedly different individual substances should be collected separately to increase disposal options and reduce cost.
6. Collection containers shall be kept securely closed except when adding hazardous material.
7. Containers to be submitted for disposal must not exceed 5 gallons (1 gallon for corrosives), unless prior EH&S approval has been obtained.
8. Secondary containment is required for all liquid hazardous wastes.
9. Hazardous wastes shall not be accumulated longer than six (6) months at satellite sites such as laboratories. The On-line Hazardous Waste tag Program assists with tracking and notifies EH&S when the accumulation of waste exceeds 180 days.

Solid Hazardous Waste Collection

1. Solid hazardous wastes must be collected into compatible containers, kept closed except when adding waste and labeled using the On-line Hazardous Waste Tag Program (http://otp.ucop.edu)
2. All sharps and broken glassware contaminated with hazardous chemicals should be placed in a puncture resistant container and sealed prior to pick-up by EH&S.
3. For handling and collection of biohazardous waste, contact EH&S at 827-5529. (http://www.ehs.ucr.edu/services/waste.html)

Collection of Gases

1. Gases will be accepted in cylinders or original shipping containers only.
2. Gas cylinders shall be equipped with a functional valve or be empty and the valve removed.
3. Whenever possible, add the name of the supplier of the compressed gas when requesting pickup through the On-line Hazardous Waste Tag. (http://otp.ucop.edu). This will aid in recycling efforts.
4. Containers shall be labeled in accordance with Section 8. Empty containers should also be marked with "EMPTY" or "MT".

Submission for disposal of collected hazardous wastes

a. To dispose of collected hazardous wastes, use the On-line Hazardous Waste Program at http://otp.ucop.edu.

20. Radioactive Waste

The radioactive waste program at UCR requires your cooperation if the large amounts of waste generated are to be handled in a way that is not only safe, but cost-effective, and otherwise responsible. Our waste management program is based on regulations related to waste disposal and the resources available at the University to optimize disposal options.

Specific requirements related to radioactive waste management include:

1. All radioactive waste must be transferred to EH&S for disposal. This means that NO radioactive material can be placed with the regular trash or poured down the drain.
2. For waste pick-up, contact EH&S Integrated Waste Management on the EH&S web site at www.ehs.ucr.edu
3. Segregate waste according to half-life and in the following general categories:
    - **Dry Solids** - All dry, solid waste must be placed in properly labeled containers provided and/or approved by EH&S. Absolutely no liquids to be included with the dry solid waste.
    - **Sharps** - All Sharps must be placed in an approved, and properly labeled, Sharps container.
        - Sharps include needles, syringes, pipette tips, broken glass, etc.
    - **Liquids** - Liquid waste includes the primary radioactive liquid and at least the first rinse.
        - Aqueous and organic waste must be collected separately and must be placed in properly labeled containers approved and/or provided by EH&S. All liquid waste containers must be compatible with their contents and stored in secondary containers large enough to contain five times the volume of the primary container.
    - **Animal/Medical Waste** - All animal/medical waste must be double-bagged, labeled, and stored in a cold room or frozen if held for more than 48 hours before pickup. Any Radioactive waste mixed with Biohazardous or Medical Waste must also comply with those requirements.
    - **Filled Scintillation Vials** - Filled vials are picked up only in flats. Segregate glass and plastic vials into separate flats.
    - **Scintillation Cocktails – Use *ONLY* Biodegradable Scintillation Cocktail fluid**

- If vials are emptied, collect the scintillation cocktails, separate from other liquids, in properly labeled containers approved by EH&S.
- Miscellaneous (high specific activity, stock vials, gels, etc.) – Contact EH&S Integrated Waste Mgt. or EH&S Radiation Safety, www.ehs.ucr.edu, for specific instructions.
- Label Information must be complete while the waste is being added and before pick-up by EH&S.
- Any Radioactive waste mixed with hazardous chemicals must also comply with the Hazardous Chemical Waste requirement and use the On-line Waste Tag Program (http://otp.ucop.edu).

- Minimize the amount of radioactive waste generated during the course of your work. Some methods to minimize waste include:
    1. Using the smallest animals possible.
    2. Using short-lived radioisotopes whenever possible.
    3. Dispose of only those items, or portions thereof, that are contaminated (cut out spots from coats, paper towels, etc.).
    4. A responsible individual must be present in the lab at the time of the pick-up.
    5. EH&S can supply some special containers for radioactive waste collection. contact EH&S Integrated Waste Management at www.ehs.ucr.edu

Appendix A - General Standard Operating Procedures (SOPs)

Integrating Safety & Environmental Responsibility into all Activities

www.ehs.ucr.edu/safety/ISEM/isem.html

Follow the five

1. Define the scope of the activity
2. Identify and analyze the hazards
3. Develop and implement controls
4. Perform the activity within the controls
5. Provide feedback and make improvements

Provide feedback & make improvements
Define scope of activity
Follow the Five!
Identify & analyze hazards
Perform activity within controls
Develop & implement controls

List of SOP's

II. Compressed Gases
III. Cryogenic Materials
IV. Peroxidizable Materials
V. Perchloric Acid
VI. Electrical Equipment
VII. CFAMM equipment
   a. Cressington 108 Auto Sputter Coater
   b. EFFA Carbon Coater
   c. FEI CM300 TEM
   d. Balzer's CPD
   e. South Bay Tech. Diamond Saw 650
   f. Gatan Dimpler 656
   g. Gatan DuoMill
   h. EDAX EDS on the XL30-FEG
   i. Lapping and Polishing Basics
   j. Gatan PIPS 691
   k. RMC Ultramicrotome MT-X
   l. South Bay Tech. Disck cutter 360
   m. FEI Tecnai12 TEM
   n. Sorvall Glass Knife Maker
   o. Cressington 308Vacuum Evaporator
   p. FEI XL30-FEG SEM
VIII. Working Alone
IX. Unattended Operations
X. Glassware
XI. Laboratory (Fume) Hood Usage
XII. Autoclaves
XIII. Hydrofluoric Acid
XIV. Formaldehyde

I. Compressed Gases

1. Define the scope of the activity
   - Handling & using compressed gas
2. Identify and analyze the hazards
   - Injury to the operator and others in the room
   - Injury to others that handle the cylinder
   - Fines for refilling containers with foreign materials
3. Develop and implement controls
   a. Engineering Controls
   - **Containers shall be stored upright and secured**. Combustible material or formed metal chains are not recommended to secure cylinders. Chains or other securing mechanisms (preferably two) should be located between one third and two thirds of the height of the container.
   - **Valves on cylinders being moved,** cylinders that are not in use, or on empty cylinders **valves shall be closed and capped.**

   b. Work Practices & Administrative Controls
   - **Compressed gases must not be transferred from one compressed gas cylinder container to another** except by the manufacturer or distributor. In some cases it is illegal to refill empty cylinders with foreign materials. Exception - refilling SCBAs from breathable air cylinders by authorized personnel.
   - **Containers shall be legibly marked** to identify the contents and give the appropriate precautionary information. (e.g. "flammable")
   - **Oxygen and oxidizing gasses shall be stored separately** from flammable and highly combustible material. Pressure reducing regulators should be used when withdrawing contents from the cylinder.
   - **Valve outlets** and pressure relief valves should be **directed away from personnel** at all times.
   - Do not exchange regulators or other appliances used with one gas with similar equipment used with other gasses. **Oils and lubricants should not be used on fittings for oxygen** or other oxidizing gasses.
   - **Do not force connections that do not fit.**

   c. Personal Protective Equipment
   - **Wear safety glasses** when using compressed gas, particularly when opening & closing valves and manipulating pressurized lines.
   - **Wear protective gloves**, such as leather, when moving compressed gas cylinders or attaching or detaching the valve caps
4. Perform the activity within the controls
   - **Follow this and other relevant procedures** when working with compressed gases
5. Provide feedback and make improvements
   - **Make note of problems** and better ways of doing things. Report these to your Laboratory Safety Officer so the procedures can be improved.

II. Cryogenic Materials

The primary hazard of cryogenic materials is their extreme coldness. They, and all surfaces they cool, can cause severe burns if allowed to contact the skin.

A. Cryogenic fluids shall be stored or handled only in containers designed for such use.
B. When personal contact with a cryogenic fluid is possible, (as when preparing cold baths or dispensing liquid nitrogen) full face shields should be worn. Wearing of watches, rings, or other items that may trap the cryogenic material should be avoided.
C. When gloves are worn while handling cryogenic materials, they should be dry, impervious and loose enough to be easily tossed off the hands. Potholders are preferred for handling cryogenic materials.
D. Lab coats should be worn over shorts and short skirts while handling cryogenic materials. Open toe shoes and sandals should not be worn.
E. Cryogenic materials should be dispensed and used in areas with good ventilation. Laboratory workers should avoid lowering their head into dry ice chests or directly over cooling baths. When transporting dry ice, or materials packaged in dry ice, the package should not be carried in the passenger compartment of the vehicle.
F. Cryogenic material may provide an oxygen-enriched atmosphere by condensing and fractionating air. This situation may increase the fire and explosion hazard of flammable and combustible materials being cooled or materials located in the vicinity of the operation.
G. Dry ice should be added to cooling baths (or liquid added to dry ice) in small increments, allowing the foaming to stop before each addition.

III. Peroxidizable Materials

A. Peroxidizable materials should be purchased in amounts that are expected to be used within six months to one year. This practice will help ensure that ethers are used up before the manufacturer's expiration date.
B. Peroxidizables, either opened or unopened, should be disposed of or tested for peroxides upon reaching the manufacturer's expiration date, or upon one year after receipt. If positive for peroxides, the peroxides may be removed or the materials may be disposed of by submitting a "Request for Chemical Pick-up" to EH&S. Please be sure to label the date that the test for peroxides was performed and the date peroxides removed (if applicable). The new expiration date will be three months after the date tested for materials in List A, and one year after the date tested for materials in List B.
C. Containers should be marked with the date opened and, in the absence of a manufacturer's expiration date, with the date received. Containers should also be marked with the date that the last test for peroxides was done.
D. Ethers should be disposed of without opening if there are visible crystals around the cap, or if the container is in a grossly corroded condition. Crystals visible in the container should be brought to the attention of the instructor, principal investigator or staff research assistant.
E. Leave at least 10% bottoms when distilling peroxidizables. The flask can be rinsed with equal amounts of a solvent such as ethanol and considered as waste.
F. Test for peroxides before distilling (even previously unopened ethers), and upon three months after opening List A and after one year of opening List B materials.

List A Peroxide hazard on storage
Test or dispose in 3 months

Isopropyl ether;
Divinyl acetylene;
Vinylidene chloride;
Potassium metal;
Sodium amide

List B Peroxide hazard on concentration

Test or dispose in 12 months
Isopropyl ether;
Diethyl ether;
Divinyl acetylene;
Tetrahydrofuran;
Vinylidene chloride
Diacetylene;
Potassium metal;
Methyl acetylene;
Sodium amide;
Dioxane;
Acetal
Decahydronaphthalene (Decalin);
Tetrahydronaphthalene (Tetralin)
Ethylene glycol dimethyl ether;
Cyclohexene;
Vinyl ethers
Diethylene glycol dimethyl ether;
Dicyclopentadiene

IV. Perchloric Acid

A. Use goggles for eye protection whenever the acid is handled.
B. In wet combustions with perchloric acid, treat the sample first with nitric acid to destroy easily oxidizable matter.
C. Any procedure involving heating of the perchloric acid should be conducted in a ventilated hood equipped with water wash down.
D. Perchloric acid hoods should be constructed of materials that are acid resistant, non-reactive, and impervious to perchloric acid, such as stainless steel.
E. Organic material should not be stored in the perchloric hood.
F. Do not allow perchloric acid to come in contact with strong dehydrating agents (concentrated sulfuric acid, anhydrous phosphorous pentoxide, etc.)
G. Perchloric acid should be used only in standard analytical procedures from well recognized analytical texts. Researchers should take the properties and hazards of perchloric acid into consideration before use.
H. If a laboratory hood or exhaust system has been exposed to perchloric acid heated above ambient temperature, tests should be conducted for explosive perchlorates before any inspection, cleaning, maintenance, or any other work is done on any part of the exhaust system or hood interior.

V. Electrical Equipment

A. General
   1. Extension cords shall not be used as permanent wiring. Power strips that are equipped with an overcurrent protection device (circuit breaker) may be used. A power strip will not be plugged into another power strip.
   2. Power cords on appliances should be inspected for damage regularly. Frayed or otherwise damaged cords should be replaced before using.
   3. To eliminate exposed wiring, outlet boxes or junction boxes shall be provided with coverplates, and receptacles shall be provided with faceplates.
   4. Ground-fault circuit interrupters should be used over sinks and in other wet areas. Ground-fault circuit interrupters should be actuated every 6 months to insure proper function.
   5. Overcurrent protection devices (circuit breakers) on panels shall be individually labeled to indicate the equipment or location of equipment served by the device.

B. Laboratory Refrigerators
   Laboratory refrigerators used for storing or cooling flammable liquids will be in compliance with NFPA 45 - Fire Protection for Laboratories Using Chemicals, section 9.2.2.2 and A.9.2.2.2. Self-defrosting refrigerators, either modified or unmodified, will not be used for storing or cooling flammable liquids.

C. Electrical apparatus
   1. Unattended electrical heating equipment should be provided with a manual reset overtemperature shutoff switch, in addition to normal temperature controls.
   2. Electric motors used to drive blenders or stirrers in open containers of flammable liquids or combustible liquids heated above their flash points should be suitable for Class I, Division 2 locations as defined in Article 500-5 of the National Electrical Code.
   3. Electrical equipment and apparatus in cold rooms should be protected from moisture due to condensation.

D. Exposed live current
   Only experienced researchers who have been trained to work safely with test instruments and equipment on energized circuits may remove enclosures and guards to perform testing on energized electrical circuits.

VI. CFAMM Equipment Standard Operating Procedures

A. Cressington 108 AUTO SPUTTER COATER

1. Place the specimen in the chamber and adjust height (we recommend about 1.5 inch or 4 cm) from the target to the surface of the sample.

2. Close the lid carefully.

3. Press the RED BUTTON on the power strip next to the pump. The pumping starts. If you hear loud noise the lid is not closed. Close it properly!

4. Adjust the time for sputtering – press and hold PAUSE/T EST button and use the up and down buttons on the SET control to adjust the time to about 60 sec., which is displayed on the right-hand side of the coater. You are free to choose different time suitable for your application, especially if the distance is different.

5. Press SET µA to check and/or set the current to 20 uA.

6. Press CYCLE to start the automatic sputtering sequence.

7. Wait for the sequence to finish. If the pumping takes too long and the CYCLE light starts to blink, turn it off and on again, so that the CYCLE light is steady on again.

8. After the CYCLE light goes off the sputtering is completed.

9. Press the RED BUTTON on the power strip next to the pump to shut down and vent the system.

10. Wait about 30 sec. Open lid and remove the specimen.

11. Close the lid carefully.

12. Record your run and the total time of sputtering in seconds in the logbook.

B. EFFA Carbon Coater Instructions

Lift the evaporation head off of the chamber and lay it upside down on the felt pad. Unravel a strand of carbon from one of the fabric squares. To install the strand(s), simply press upwards against the spring-loaded cap screws. Place the carbon strand in the gap between the carbon discs and release the spring. The strands are held in place by spring pressure. If two or more strands are used, they may be twisted together three or four times before clamping into the second electrode. Handle the carbon strands carefully, as they are easily frayed.

When the vacuum the dial on the approximately 200µ, switch on the output and turn up the dial on the powerstat. The carbon strand will glow a dull orange, increasing to a brilliant white as the power is turned up. The dial should quickly be turned up until the carbon burns out. This will take only three or four seconds. It will flash and go dark, much like a light bulb. This is when most of the carbon evaporation occurs. In the course of the evaporation, the vacuum will normally drop off to about 250µ. Do not increase the power too slowly or let it reside at a low setting as this will only generate a lot of excess heat. Damage to the sample and/or chamber may result.

As soon as the evaporation is completed, switch off the Output and Main switches. Return the powerstat to zero, bleed the chamber and remove the sample. The carbon strand will have burned out into two pieces, which are discarded.

Because there is no isolation valve for the chamber, it is suggested that the chamber not be left under vacuum when not in use. Many low-cost vacuum pumps do not have anti-suction valves. Leaving them under vacuum when shut off can cause the oil to be drawn up the hose to the chamber.

Sometimes the ends of the carbon strands will continue to arc or flicker after burning out. This can cause carbon participates to fall on the sample and is avoided by quickly switching off the output.

The carbon film thickness can be varied in increments by using one, two or three strands at a time. Using more than three strands will overload the output and is not recommended. Substituting optional chamber sections of different heights varies the sample distance.

After a number of evaporations, carbon dust and fiber fragments will begin to accumulate in the chamber and on the gaskets. These require an occasional wiping with a cloth. Carbon will also build up on the posts and the underside of the head and will eventually begin to flake off. These should be kept free of loose carbon. The chamber can be cleaned with common lacquer thinner, which does not attack Lucite

An alternate method of evaporation is as follows: After the carbon is loaded and the system is pumping, preset the powerstat to about half-scale, or ''50''. When the vacuum reaches about 150µ, switch on the Output. The power surge will cause the carbon to burn out in about one second. This method will yield almost twice as much carbon per strand as will slowly turning up the dial. However, it also tends to generate participates which become visible at higher magnifications. Under 5,000X, there appears to be no difference in the carbon coatings whether they are done quickly or slowly. In the case of a heat-sensitive sample, the faster a coating can be deposited, the better. Remember to switch off the Output, or it will re-energize as soon as the vacuum switch closes on the next run.

The EFFA Carbon Coater can also be used for making carbon or carbon-stabilized substrates on TEM grids. This requires a vacuum of 60µ or better. This in turn requires dean gaskets and a 50-liter/minute pump. Secure the grids to a glass microscope slide. Place the slide directly on the sample platform. Use the standard 3'' chamber sections and two strands of carbon. To evaporate, dial the power up and down five or six times, or until the strands burn out. This minimizes heating and outgassing. Carbon films made this way show no structure at up to 240,000X.

C. FEI CM300 TEM

HANDLING SF_6 GAS

-SF_6, Sulfur hexafluoride gas, is colorless, odorless, non-flammable and non-toxic (if not heated above 250 °C).

- The gas is heavier than air.

- The gas causes suffocation at high concentration levels.

- The gas breaks down when overheated above 250°C giving off highly toxic fluorine gas.

THEREFORE:

- The ventilation system must be switched to extract AT ALL TIMES.

- Smoking in SF_6 sensitive areas is FORBIDDEN.

- THE SF_6 DETECTOR MUST ALWAYS BE IN OPERATION.

- GAS MASKS (2X) AND RUBBER GLOVES (2 PAIRS) MUST BE PRESENT.

- FOR TRANSPORTING THE GAS, USE THE TOOLS AND EQUIPMENT DELIVERED

WHAT TO DO IN EVENT OF A SF_6 LEAKAGE

SWITCH OFF ALL THE HEAT SOURCES.
MAKE SURE THE VENTILATION SYSTEM IS WORKING,
LEAVE THE ROOM,
CLOSE THE DOOR, AND
NOTIFY THE CFAMM STAFF.

1. CM300 Start up

*Do not start if you haven't read thoroughly at least once sections **1, 2, 4, 6,** and **7** of the CM300 Operating Instruction manual!*

*If the whole system is down go to **COLD START** (next section on this page).*
*If the CM300 is operating but the **STANDBY** button is not illuminated go to **START FROM STANDBY MODE** (section 3 on this page).*
If the CM300 is operating and

*MICROSCOPE **ON** button is not illuminated,*
***STANDBY** is illuminated,*
*MICROSCOPE **OFF** button is illuminated red,*
*If **HT** button LED is not illuminated go to section 3.1 **Starting High Tension** (p.3).*
*If **HT** button LED is illuminated go to **Inserting the specimen** (p. 5) and continue with p.6.*

2. Cold start

1. *Turn on the water and pneumatic supplies to the microscope if*
   air pressure is in the range 70 psi to 90 psi;
   water temperature ~ 62 F (16° C ±1° C);
   room temperature ~ 68 F (20° C ±3° C).
2. *Switch on the electrical supply to the microscope.*
   *MICROSCOPE **STANDBY** button will illuminate.*
3. *Press MICROSCOPE **ON** button.*
   *MICROSCOPE **OFF** button will illuminate red.*
4. *Continue with next section.*

3. Start from Standby mode

*CM300 is in standby mode when the **STANDBY** button is not illuminated.*

1. *Press **VACUUM SYSTEM ON** button.*

VACUUM SYSTEM OFF *button illuminates green.*

2. *Wait! Normally about 30 min. Operational vacuum is reached when the UHV indicator light comes on.*
3. *Continue with* ***Starting High Tension*** *(p. 3).*

3.1 Starting High Tension (HT)

1. *Select* ***MODES*** *->* ***TEM*** *this brings you to the* ***TEM BRIGHT FIELD*** *page,*
2. *Select focus step* ***6*** *using the* ***STEPSIZE*** *knob on the right-hand side panel*
3. *Go to* ***PARAMETERS*** *page, select* ***HT*** *of 50 kV.*
4. *Select* ***EMISSION 1****.*
5. *When vacuum of* ***IGP*** *reaches* ***≤30*** *at HT ≤ 100 kV or* ***≤27*** *above 100 kV,*

   press ***HT*** *button, green light above* ***HT*** *button comes on.*

   Wait until ***HT*** *reading indicates 50 kV*

   (on ***PARAMETERS*** *page above the* ***IGP*** *reading)*

3.2. Selection of High Tension (HT)

for HT ≤250 go to step 3.2.1.;

for HT of 250 or 300 kV go to step 3.2.2;

for HT ≥250 but not multiple of 50 go to step 3.2.3.

3.2.1. Setting HT ≤ 250 kV.

1. *If the desired HT is ≤ 250 kV and multiple of 50 select the desired HT using the softkeys on the* ***PARAMETRS*** *page. After the desired voltage has been reached proceed with* ***Filament saturation*** *(p. 7). If the desired HT is ≤ 250 kV and not multiple of 50 go to the next section.*
2. *Selecting HT that is ≤250 kV and not multiple of 50.*

   1. *Go to the TEM BRIGHT FIELD page*
   2. *Adjust the STEPSIZE knob to an appropriate value: Step size* ***7*** *for 5 kV per step, step size* ***6*** *for 2.5 kV per step, step size* ***5*** *for 1.25kV per step etc.*
   3. *Go back to the* ***PARAMETRS*** *page.*
   4. *Using the* ***HT*** *softkeys select HT closest to but less than the desired final HT.*
   5. *Press* ***FREE CONTROL***
   6. *Start turning the* ***FOCUS*** *knob clockwise to increase the HT.*

   Wait about a second after each click of the knob and observe the Emission current, and the IGP vacuum level. If emission reading increases more than about 5 scale readings or the IGP readout increases wait until they settle down before you continue with the next click.

   7. *Stop when the desired HT is reached (reading on* ***PARAMETERS*** *page under the* ***IGP*** *reading). Do not deselect* ***FREE CONTROL****.*

*8. Proceed with **Filament saturation** (p. 7).*

3.2.2. Setting HT of 250 or 300 kV

*1. Go to the **TEM BRIGHT FIELD** page*

*2. Adjust the **STEPSIZE** knob to focus step **6** (2.5 kV) or less.*

*3. Go to the **PARAMETRS** page and select HT of 250 using the HT softkeys.*

*Wait until HT reading indicates 250 kV (on **PARAMETERS** page below the **IGP** reading).*

*4. Press the **CONDITIONING** softkey.*

*5. Start turning the **FOCUS** knob clockwise and delay for about two seconds after each click. If **EMISSION** reading increases more than about 5 scale readings or the IGP readout increases wait until they settle down.*

*Continue until you reach HT value of 10% above the desired final operating voltage (reading on **PARAMETERS** page, right above the **IGP** reading).*

6. Leave the microscope to settle down at these conditions for about 15 min.

*7. On the **PARAMETERS** page press the **CONDITIONING** softkey, HT returns to the desired operating voltage.*

3.2.3. Setting HT ≥250 kV and not multiple of 50.

*1. Adjust the **STEPSIZE** knob to focus step **6** (2.5 kV) or less.*

*2. Go to the **PARAMETRS** page and select HT of 250 using the **HT softkeys**.*

*Wait until HT reading indicates 250 kV (on **PARAMETERS** page above the **IGP** reading).*

*3. Press the **FREE CONTROL** softkey.*

*4. Start turning the **FOCUS** knob clockwise and delay for about two seconds after each click. If **EMISSION** reading increases more than about 5 scale readings or the **IGP** readout increases wait until they settle down.*

*5. Stop when you have reached HT value of 10% above the desired final operating voltage (reading on **PARAMETERS** page, right below the **IGP** reading).*

*6. After about 15 min. decrease HT voltage to the desired setting using the **FOCUS** knob.*

*7. Stop when desired HT is reached (do not deselect **FREE CONTROL**!).*

*8. Proceed with **Filament saturation** (p. 7).*

4. Inserting and removing a specimen

IMPORTANT:

Insert the specimen holder before saturating the filament.

*Select **MODES -> CONFIGURATION**.*

*If **ACTUAL** under **FILAMENT HEATING** is 0 insert the specimen following the steps below.*

*If **ACTUAL** is at max. value decrease saturation by 5 steps using the **FILAMENT** control knob and saturate the filament again after inserting the specimen holder and when the vacuum of **IGP** reaches **≤30** at or below 100kV or **≤27** above 100kV .*

1. *Place and secure the specimen grid (3.05 mm diameter) in the specimen holder.*
   Do not touch with bare hands the part of the specimen holder, which goes into the column (from the tip to the rubber O-ring).
2. *Grab the holder by the black round handle*
3. *The specimen holder is introduced into the column so that the small pin on the side of the holder coincides with the slit on the **Compustage** where it reads **CLOSE**.*
4. *Insert the holder half way into the column until it stops, the red light on the Compustage comes on.*
5. *The control screen changes to **SPECIMEN HOLDER SELECTION**.*
   *Select the holder type by pressing the corresponding softkey on the CM300 control screen page and press **READY** button.*
   Connect the holder cable if working with the double-tilt holder.
6. *Wait until the specimen chamber is evacuated and the red light on the Compustage goes off.*
7. *By holding the black handle rotate the holder counterclockwise until stop (~ 120°) and hold it firmly.*
   The vacuum will try to suck the holder in. Gently release the grip to allow the holder to slide all the way into the microscope column
   *Proceed with **Overnight Standby Mode** (p.6) or **Filament saturation** (p.7)*

Removing a specimen

1. *Decrease the **FILAMENT** saturation by about 5 steps by turning the FILAMENT knob counter-clockwise and delaying for about 1 sec at each step.*
2. *Go to **TEM BRIGHT FIELD** ->**COMPUSTAGE** -> **COMPUCTRL** press **RESET HOLDER**. By holding the black handle pull the holder straight back halfway out until stop, turn it clockwise until stop (~ 120°) and pull it completely out.*

5. Start from Overnight Standby Mode

*If **HT** button LED is not illuminated go back to section **3.1**.*
*In case the **HT** button LED is illuminated continue with the steps below.*

5.1.Selection of High Tension (HT)

1. *Go to the **TEM BRIGHT FIELD** page*
2. *Adjust the **STEPSIZE** knob to focus step **6** (2.5 kV) or less.*
3. *Go to the **PARAMETRS** page and verify that the select HT is 250 or 300 kV*
4. *Select **EMISSION 1**.*

for HT multiple of 50 go to step 5.

for HT not multiple of 50 go to step 6.

*5. If the desired HT is multiple of 50 select the desired HT using the softkeys on the **PARAMETRS** page. After the desired voltage has been reached proceed with **Filament saturation** (p. 7).*

If the desired HT is ≤ 250 kV and not multiple of 50 go to the next section.

6.Selecting HT not multiple of 50.

*6.1. Go to the **TEM BRIGHT FIELD** page*

*6.2. Adjust the **STEPSIZE** knob to an appropriate value: Step size **7** for 5 kV per step, step size **6** for 2.5 kV per step, step size **5** for 1.25kV per step etc.*

*6.3. Go back to the **PARAMETRS** page.*

*6.4. Using the **HT** softkeys select HT closest to but less than the desired final HT.*

*6.5. Press **FREE CONTROL***

*6.6. Start turning the **FOCUS** knob clockwise to increase or decrease the HT.*

Wait about a second after each click of the knob and observe the Emission current, and the IGP vacuum level. If emission reading increases more than about 5 scale readings or the IGP readout increases wait until they settle down before you continue with the next click.

*6.7. Stop when the desired HT is reached (reading on **PARAMETERS** page under the **IGP** reading). Do not deselect **FREE CONTROL**.*

*6.8. Proceed with **Filament saturation** (p. 7).*

6. Filament saturation

***IMPORTANT**:*

Insert specimen holder (go to page 5) before saturating the filament.

After inserting the specimen holder proceed only if vacuum of

*of **IGP** reaches ≤**30** at or below HT of 100kV or ≤**27** above 100kV .*

*1. On the **TEM Bright Field** page select **MODES***

*2. Select **CONFIGURATION***

***FILAMENT HEATING** should have **FIL LIMIT** highlighted*

***CATHODE** should should have **LaB_6** highlighted*

*3. If **FIL LIMIT** is highlighted go to the next step.*

*If **FIL LIMIT** is not highlighted stop here!!!*

Do not proceed further and contact staff personnel.

4. Turn the **FILAMENT** knob on the right-hand side panel several times clockwise to heat up the filament up to the value of the **FIL LIMIT**. The CM300 automatically delays 5 sec. between each step. It will beep when the preset **FIL LIMIT** is reached and the filament is saturated.
5. Proceed with **Obtaining a Bright Field image** (p. 8).

7. Obtaining a Bright Field image

1. Go to **MODES SELECTION** page
2. Select **DET CONF**

   USER SELECTION should read **AUTO**

   TV-SYST should read **CENTRAL**
3. Go to the **TEM** page, select magnification in the lower **M** mode (1800 - 3000x) with the **MAGNIFICATION** knob.
4. Select spot size **2** using the **SPOT SIZE** knob. Make sure **objective** and **SA** apertures are removed from the beam (lever to the right).
5. Using the **INTENSITY** control knob on the left-hand side panel, adjust the beam intensity so that the entire fluorescent screen is illuminated. If the light on the screen is too dim go to the **PARAMETERS** page and increase the emission current setting using the **EMISSION** softkeys, Do not exceed 20 μA (reading on the **EMISSION** meter).
6. Selecting and centering of the **C2** aperture

   6.1. Select desired **C2** aperture by turning the large selector knob (top aperture holder on the microscope column).

   6.2. Turn the **INTENSITY** knob to focus the beam to the smallest possible spot.

   6.3. Center the spot using the **X-Y SHIFT** knobs on the right-hand side panel.

   6.4. Turn the **INTENSITY** knob clockwise to spread the beam so that the illuminated circle is just smaller that the big fluorescent screen.

   6.5. Center the illuminated circle using the **C2** aperture mechanical shift controls (top aperture holder on the microscope column).

   6.6. Repeat 6.2. to 6.5. until beam remains centered after focusing and spreading it.
7. Focus image

   7.1. Go to **TEM BRIGHT FIELD** page, select appropriate focus step by using the **STEP SIZE** knob

   7.2. Obtain lowest contrast by turning the **FOCUS** knob

   7.3. If not sure about the focus.

   Press **WBL** button on the left-hand side panel. If not in focus the image appears split. By turning the **FOCUS** knob make the splitting disappear.

8. Adjust the eucentric position of the specimen

8.1. Select magnification between 2000 - 5000x

*8.2. Adjust beam intensity on screen (**INTENSITY** knob, **SPOT SIZE, SHIFT**)*

*8.3. On the specimen select easy recognizable feature with good contrast and bring it to the center of the fluorescent screen using the **XY** joystick and focus using the **FOCUS** knob*

*8.4. Go to the **COMPUSTAGE REGISTER CONTROL** page, press **A-WOBBLER***

8.5. Observe the movement of the selected feature on the screen if not eucentric the feature moves across the screen.

*8.6 Reduce the amplitude of movement by using the **Z**-joystick lever.*

*8.7 Press **A-WOBBLER** again to deselect it.*

*8.8 Press **READY** to go to the main **TEM** page.*

9. Check beam alignment

*/see section 2.6 in the **CM300 Operation instructions** manual p. 2-60 to p. 2-61.*

If necessary carry out the following Direct Alignments:

***GUN SHIFT** /see p. 2-67 in CM300 Operating instructions/*

***ROT CENTER** /see p. 2-68 " " "/*

***PIVOT POINT X, Y** / see p. 2-66 " " "/*

***IMAG SHIFT** / see p. 2-67 " " "/*

10. Check image astigmatism

*/section 2.5 in the **CM300 Operation instructions** manual p. 2-47 to p. 2-59/.*

If proper alignment cannot be achieved after caring out the above procedures contact the CFAMM staff.

11. Selecting and centering the objective aperture

*11.1. Insert **SA** aperture #1 by swinging the lever of the SA aperture holder to the left (lowest aperture holder on the column).*

*11.2. Place the **SA** aperture over a region transparent for the electrons using the mechanical shift controls of the SA aperture holder.*

*11.3. Press the **D (ZOOM)** button on the right-hand side panel, SA electron diffraction pattern appears on the fluorescent screen.*

*11.4. Using the **MAGNIFICATION** knob select camera length (**D**) between 400 - 800 mm. Focus beam in a spot using the **FOCUS** knob*

11.5. Insert the desired objective aperture by swinging the lever of the objective aperture holder to the left (middle aperture holder on the microscope column).

11.6. Center the objective aperture around the central transmitted spot using the mechanical shift controls of the objective aperture holder.

*11.7 Press the **D (ZOOM)** button to switch back to imaging mode*

*11.8 Take out the **SA** aperture by swinging the mechanical lever to the right.*

*12. Go to **TEM** page, select desired magnification by turning the **MAGNIFICATION** knob.*

*13. Select appropriate spot size using the **SPOT SIZE** knob*

*Adjust beam intensity using the **INTENSITY** knob and center illumination using **X-Y SHIFT** knobs.*

14. Focus image.

15. Ready to record image.

8. Recipe for SAED Patterns

1. Decide on the area from which you wish to obtain diffraction pattern.

*2. Ensure that this area is at eucentric height, by selecting the "**A-WOBBLER**" softkey on the compustage page. The area should remain stationary during rocking. Deselect "**A-WOBBLER**".*

*3. Defocus condenser lens using "**INTENSITY**" knob so that the whole fluorescent screen is illuminated evenly.*

*4. Insert **SA** aperture so that it surrounds the area of interest. Change the aperture if the size does not fit your area.*

*5. Push "**D**"(**ZOOM**) button for diffraction.*

*6. Defocus the central transmitted spot so that you can see the image in the spot. Check if the area you selected is in the central spot, if displaced bring it in the spot using **X** and **Y** joystick controls.*

7. Select appropriate camera length using magnification control.

8. Focus central transmitted beam to the finest possible spot using the focusing knob.

9. To record the pattern block the strong central transmitted spot with the mechanical pin and then record pattern on the computer using the CCD camera.

9. Taking photographs

1. Obtain an image on the fluorescent screen.

*2. Position detail of interest using the **XY**-joystick within the marks on the fluorescent screen.*

*3. Select **TEM CAMERA** page.*

4. Select exposure parameters.

*5. Introduce the small fluorescent screen if using **AUTO** exposure.*

*6. Lift the main fluorescent screen handle, the green light on the **EXPOSURE** button comes on.*

*7. Make sure that the **SHUTTER** on the **GATAN CCD** camera control box is in **OPEN** position or the control box is switched off.*

8. Put the protective rubber cover over the viewing glass or turn all lights down.

*9. Press **EXPOSURE** button, green light goes off.*

*10. Green light on **EXPOSURE** button comes on, when exposure is done.*

Lower the main fluorescent screen.

10. Digital Image Acquisition

*1. Login on the PC computer and launch **DigitalMicrograph**.*

*2. Turn on **GATAN CCD** camera [control box **RETRACTABLE MULTISCAN CAMERA** on the left side of the TV monitor].*

*3. Set the **PELTIER COOLER** [control box **RETRACTABLE MULTISCAN CAMERA**] switch to **COOL** position. Images can be acquired after the CCD has been cooled for at least half an hour.*

4. After the CCD has been cooled for at least half an hour collect gain reference images.

*4.1. Remove the specimen from the el. beam. Using **INTENSITY** knob spread the beam so that the image of the beam is as big in diameter as the big fluorescent screen.*

4.2. Lift the main fluorescent screen using the lever on the left hand-side of the viewing chamber.

*4.3. Set **CAMERA** switch to **COMPUTER** position [control box **RETRACTABLE MULTISCAN CAMERA**].Switch **SHUTTER** control to AUTO position [control box **RETRACTABLE MULTISCAN CAMERA**]*

*4.4. In **DigitalMicrograph** on the computer screen under pull down menu **CAMERA** with the left mouse button select **INSERT CAMERA**.*

*4.5. Under pull down menu **CAMERA** select **PREPARE GAIN REFERENCE** and follow the instructions on the computer monitor [see DigMic User's Guide for more details].*

*5. After the gain reference has been collected insert the specimen under the el. beam. Lower the main fluorescent screen. Obtain an image and bring the feature of interest in the middle of the main fluorescent screen using the **XY**-joystick controls.The area, which can be imaged with the CCD is slightly smaller than the area which is viewed on the small fluorescent screen.*

6. Lift the main fluorescent screen using the lever on the left hand-side of the viewing chamber.

*In **DigitalMicrograph** on the computer screen under pull down menu **CAMERA** with the mouse select **INSERT CAMERA***

*7. In DigitalMicrograph on the computer screen in the **CAMERA VIEW** panel under **SETUP** select **SEARCH.** Click on the tools icon select **FULL CCD** and **BINNING 2**. Set exposure time. Click OK to close the tools window.*

*Click **START VIEW**, one can adjust the scanning time by using the **UP** or **DOWN** arrow keys on the computer keyboard.*

Adjust image parameters.

8. *Image acquisition*

   *8.1. In the **CAMERA Acquire** panel click on the tools icon, select **FULL CCD**, under **SETUP** select **RECORD**, under **BINNING** select **1**, Under **PROCESSING** select: **FRAMES:** 1, **CORRECTIONS**: **GAIN NORMALIZED**. Set desired exposure time. Close the setup window.*

   *8.2. Under **FILE** menu select **Global Info** and **Scale Bar**, in the **Scale Bar** panel check the box if you want scale marker automatically placed on your acquired images.*

   *8.3. In the **CAMERA ACQUIRE** panel click on the **START ACQUIRE** button to acquire the image. [see in DigitalMicrograph User's Guide for details about setting up parameters].*

9. *The acquired image can be saved to disk in*

   DM3 GATAN format [usable only with DigitalMicrograph],

   in TIFF or JPG format for more details see DigMic User's Guide.

11. Imaging with TV camera

1. *Switch on the **TV** camera control box on the left-hand side of the microscope column and wait about 20 sec for the green light of the **SIGNAL LEVEL** indicator /on TV camera control box/ to flash for a short period.*
2. *Check the settings on the TV control unit*

   *- **SHADING CORRECTION** and **FILTER** should be **ON**,*

   *- **BRIGHTNESS**, **CONTRAST** and **INTENSIFIER** should be in **AUTO***

3. *Switch on the TV monitor /monitor on the right hand side of the microscope column/.*
4. *Set the button on the lower left side of the TV monitor to **A** position.*
5. *Bring the feature of interest in the middle of the fluorescent screen using the joystick controls. Only the area under the small fluorescent screen is visible on the TV monitor.*
6. *Adjust intensity of the beam so that the whole area of the main fluorescent screen is illuminated. Lift the main fluorescent screen [lever on left-hand side of the viewing chamber]. Retract the CCD camera if necessary.*
7. *In couple of seconds the image will appear on the TV monitor.*

   *Adjust the el. beam intensity **INTENSITY** knob on the left-hand side microscope panel] to optimize the image.*

12. Ending the TEM session

1. *De-saturate the filament by turning the **FILAMENT** knob counter-clockwise and delaying for about 1 sec. between individual click steps until it beeps or **ACTUAL FILAMENT HEATING** on the **MODES** -> **CONFIGURATION** page reaches **0**.*
2. *Set magnification with the **MAGNIFICATION** knob in the **M** mode range (~3000x).*

*3. On the **TEM** -> **PARAMETERS** page set **EMISSION** to **1**.*

*4. **DO NOT** switch off the accelerating voltage.*

If HT is at 300 kV leave it there.

*If HT is less than 300 kV bring the HT to 250 kV using the **HT** softkeys on the **PARAMETERS** page.*

*5. Go to **TEM BRIGHT FIELD** ->**COMPUSTAGE** -> **COMPUCTRL** press **RESET HOLDER**.*

6. By holding the black handle pull the holder straight back halfway out until stop, turn it clockwise until stop (~ 120°) and pull it completely out.

7. After removing the specimen grid from the holder put the holder in its support and store it in the desiccator box.

8. Log out from your account on the PC computer, if logged in. Do not shut down the PC.

STOP HERE!

Fill out the log book.

Do not continue further without special permission from the CFAMM staff.

13. Shutting down CM300 (CFAMM STAFF ONLY!!!)

*1. De-saturate the filament by turning the **FILAMENT** knob counter clockwise and delaying for about 1 sec. between individual click steps until it beeps or **ACTUAL FILAMENT HEATING** on the **CONFIGURATION** page reaches **0**.*

*2. Press **HT** button to switch off the accelerating voltage.*

*3. Press the **STANDBY** button.*

STOP here for Standby mode.

4. Wait until any automatic pumping sequence is completed.

*5. Press the **MICROSCOPE OFF** button.*

6. Switch off the electrical supply to the microscope.

7. After approximately 30 min., turn the water supply off.

8. Turn off the pneumatic supply.

9. Turn off the Nitrogen gas supply, if in use.

Electronic sign-up

1. You will receive an email with your user name and password.

2. Using a web browser go to http://faces.ccrc.uga.edu/

3. Login by typing:

CFAMM in the GROUP field,

in the USER NAME field type in your logon name,
in the PASSWORD field type in your password
Click the GO button.

4. *Under "select a resource", choose the instrument you want to sign-up for, in your case "CM300"*
5. *By clicking the month and dates, choose the date you wish.*
6. *Click the button to the right of the time you wish to start, then click the button to the right of the time you wish to end.*
7. *Click the "reserve this time" button when you have chosen the date and times you desire.*
8. *Confirm this in the box that comes up.*
   Do not leave messages for in the space available, email bozhilov@ucr.edu directly if there is an issue that needs attention.
9. *Logoff - you are scheduled. You can reschedule if you need to - follow the above directions, at #6 click on the button for the time you have already scheduled (the date must already be set appropriately), you will be given the option to cancel the appointment.*

NOTE- IF YOU CANCEL AN APPOINTMENT LESS THAN ONE WORKING DAY IN ADVANCE, YOU WILL BE CHARGED FOR 2 HOURS OF USE.

D. Balzer's Critical Point Dryer

Drying and Cooling gas is CO_2.

Transferring liquid may be acetone, ethanol. Freon.

Approximately 7 runs per tank of CO_2

1. Check the log book for number of runs -- log in.

Load

2. Make sure all three valves are closed (gas inlet, gas outlet and gas metering).
3. Unscrew the chamber lid. If there is pressure in the chamber (lid won't unscrew) open both the gas valves, then close again.
4. Check to make sure that the magnetic stir bar and serrated disc are in place.
5. Fill the chamber both enough of the transferring liquid (acetone, etc.) so that the specimens within the transfer baskets will be covered.
6. Rapidly transfer the sample lid down, hand tight only.
7. Turn on the Main switch and open the valve on the CO_2 tank all the way.

Cool

8. Set the chamber cooling temperature to +15 ° C.

9. Depress the TEMP button. The CPD will began automatically cooling the specimen chamber to the selected temperature. When 15 ° C is reached exchange of the transferring liquid (acetone) with the drying gas (cm CO_2) can begin. The CPD will maintain a temperature of +15 ° C, + or – 3°.

Exchange

10. Slowly open the gas inlet valve. Observe via the sight glass the filling of the chamber. Note the Schlerin line patterns. These indicate the mixing of fluids and signals the beginning of exchange.

11. When the chamber is nearly full close the Inlet valve. Depress STIRRER button. Let the fluids mix far 3-4 minutes. Do not use the magnetic stirrer when fragile specimens are to be dried.

12. Switch off the stirrer.

13. Open the gas out valve one or more turns. Slowly open the metering valve and drain the liquid from the chamber observing the float level through the sight glass.

14. When the fluid level is lust above the specimen basket (or the specimens inside the basket); close the metering and gas nut valves.

15. Repeat steps 10-13 until all traces if acetone is gone and until a fine spray of dry CO_2 is emitted during the surge.

Heat

16. Fill the chamber almost to the top slight glass. To prevent damage to the burst membrane do not fill the chamber completely.

17. Make sure all valves on the CPD are closed. Shut off the magnetic stirrer. Close the CO_2 valve all the way.

18. Set the temperature to 42 ° C. The CPD will automatically began warming the chamber. The pressure within the chamber wall also began to rise. Monitor the pressure carefully. If the pressure approaches 1500 psi, reduce the pressure both the gas nut and metering valves to 1160-1230 psi. At 1600-2000 psi, the burst membrane will rupture and your samples will be ruined.

19. As the critical temperature and critical pressure are approached. the CO_2 will go from a liquid to a gaseous state. If there has been an inadequate exchange CO_2 for the acetone in your sample, the pressure will not exceed 1000 psi, in which case the critical point is never reached. Samples may be salvageable, so proceed with pressure reduction.

20. To ensure complete drying has taken place, wait until the critical temperature and pressure are exceeded:

    Critical values of CO_2 are 31.0 ° C; 1072 psi;
    so go to 40 ° and 1160-1230 psi.

21. Begin pressure reduction by opening the gas out valve one or two turns and slowly

open the metering valve. The metering valve allows you to regulate the rate of pressure reduction which should take 15-20 minutes. If the gas within the chamber begins to recondense, you are reducing the pressure too quickly.

22. At 0 psi, fully open the gas put and metering valves. Unscrew the chamber lid and remove you samples. Samples will be very hygroscopic.

23. Replace the lid but don't screw on the lid. Close all valves, release the temperature button, and Shut off the main valves.

FINISH

E. South Bay Tech Diamond Saw 650

3.3: Standard Operating Procedure

The following procedure is a basic guideline for cutting specimens using the Model 550. Although certain aspects of the procedure will be modified to fit a particular application, the basic operation is still the same.

3.3.1: Mounting the Diamond Wheel

1. Adjust the down stop of the work arm assembly such that the down stop is extended fully towards the switch.
2. Place the first spacer on the shaft and push against the shaft support.
3. Place the first diamond wheel support collar onto the shaft and slide flush with the wheel spacer.
4. Place the diamond wheel onto the shaft and slide flush with the diamond wheel support collar.
5. Slide the next diamond wheel support collar on, followed by the final wheel spacer.
6. Place the retaining screw on the end of the shaft and tighten.

NOTE: The wheel can be oriented in any position along the shaft, as long as the same number of spacers and collars are used.

3.3.2: Setting the Load

1. Adjust the down-stop to the fully extended position. This prevents the work arm from slamming down onto the diamond wheel.
2. Mount the desired specimen holder into the sample holder locking screw assembly.
3. Remove the cutting weights from the work arm assembly.
4. Rotate the counter-weight counter-clockwise until the arm is balanced above the drive shaft of the Model 650.
5. Apply the desired amount of cutting weights to the work arm for the applied load.
6. To increase the load, adjust the counter-weight position by turning clockwise.

3.4.3: Setting the Downstop

Setting the down-stop will determine when the instrument terminates a cutting process. The down-stop also helps prevent damage to the diamond wheel when calibrating, mounting, and applying load before processing.

1. Make sure the diamond wheel is installed.
2. Place a mounted specimen into the desired sample holder.
3. Loosen the slide assembly locking screw and adjust the position of the specimen such that the diamond wheel edge is approximately 5mm away from the edge of the specimen.

Note: Keep the work arm assembly slightly above the diamond wheel. DO NOT allow the work arm assembly to slam down on the diamond wheel. Damage will occur.

4. Loosen the locking nut of the down-stop. Hold in position and adjust the down-stop to the desired height.

5. Gently lower the work arm assembly onto the diamond wheel and verify the position. Re-adjust the position until it is set properly.
6. Hold the down-stop in place and tighten the locking nut of the down-stop to set the position.
7. Raise the work arm assembly and slide into the proper position where the specimen will be cut.
8. Tighten the slide assembly locking screw to lock in position.

3.4.4: Cutting a Specimen

Checklist

1. Be sure that the micrometer is installed properly. See Section 2.4.2
2. Be sure that the wheel is mounted properly. See Section 2.4.3
3. Be sure the proper sample holder has been installed.
4. Check that the instrument is plugged in.

Procedure

1. Place coolant (diluted to 30 parts water; 1 part coolant by volume) into coolant tray until the coolant covers ~ 10-15 mm (1/2") of the diamond wheel. Do not overfill.
2. Install the coolant tray into the Model 650.
3. Align the specimen in the specimen holder so that the specimen is positioned on the holder in the desired orientation to the cutting wheel.
4. Loosen the slide assembly locking screw at the rear of the instrument and roughly align the area to be cut onto the cutting wheel by moving the slide assembly.
5. Tighten the slide assembly locking screw and finely adjust the specimen position using the micrometer.
6. Adjust the cutting load as described in Section 3.3.2.
7. Adjust the down-stop position as described in Section 3.3.3.
8. Turn the main power switch ON by depressing the switch.
9. Push the START button on the front panel to start the motor.
10. Adjust the speed using the speed control knob on the front panel. Maximum speed is used in many cases.
11. Dress the diamond wheel with a SiC dressing stick manually or by using the Model 65012 Dressing Stick Holder.
12. Gently lower the arm with the specimen onto the diamond wheel to begin cutting of the specimen.

CAUTION: Do NOT allow the arm to slam down onto the cutting wheel. Severe damage to the wheel will result.

13. Adjust the speed and load as needed.

14. When cutting is complete, raise the arm into the up position and turn the main power switch OFF.
15. Remove specimen.

F. GATAN Dimpler 656

STEP 1. PREPARING THE SPECIMEN

Note: Specimens must have perfectly parallel faces prior to dimpling!!

a) Make standard thin section for TEM (thickness 50~100 um) with surface finished on a polishing cloth with 0.05 um alumina suspension;
b) Search target under the optical microscope and glue a mesh grid on it; use as less epoxy as possible; (Alternatively, you may cut a disc to dimple first and glue the grid afterwards.)
c) After curing, place the thin section on the hot plate and take off a piece with the grid;
d) Soak in acetone for 5 mins to get rid of wax on surface;
e) Cut carefully a perfect 3mm diameter disc with sharp lmife along the edge of the grid under the binocular microscope, measure and record its initial thickness with the grid;
f) Put a transparent specimen mount on the hot plate, melt a small quantity of wax on the end;
g) Carefully place the specimen on the mount, polished surface (with grid) down;
h) Gently press the specimen onto the mount and move in small circles to ensure that the wax evenly and thinly distributed. Avoid getting wax on the upper surface and bubbles on the bottom surface of the specimen;
i) Remove the mount from the hot plate and allow to cool;
j) Use acetone and a cotton bud to gently remove excess wax from the specimen mount;
k) Check the thickness of the wax layer. It should not be more than 5 um;
1) Grind the specimen thickness (with grid) to 70~100 um with proper jig (e. g. 623 Disc Grinder);

Recommendation: 40urn (grit)---> 160 um (thickness); 15 um ---> 120 um; 5 um ——> 100 um

STEP 2. DIMPLER POWER ON:

STEP 3. DIAL INDICATOR ZERO:

Note: The zero of the dial indicator should always be checked prior to dimpling. The care with which the zero is set will determine the accuracy of the measuring systems.

a) With the arm in the upright position, remove the specimen mount from the magnetic turntable;
b) Lower the arm onto the cam;
c) Check that both motors are off and set a load of 20 g;
d) Lower the micrometer drive (clockwise) until it bottoms against the base;
e) With the cam, lower the arm;

f) Raise the micrometer (counter-clockwise) until the dial indicator needle has rotated just over one complete turn and comes to rest at or near the 12 o'clock position;
g) Rotate the face of the dial indicator until the zero is positioned exactly under the needle, taking care to avoid parallax errors. Pressing down on the arm to adjust the dial indicator will not cause any damage, provided the force is not excessive, but structural flexing will produce a small change in the needle position;

Note: It is not necessary for the needle to be exactly at the 12 o'clock position.

STEP 4. SPECIMEN POSITIONING: The magnetic specimen turntable and light microscope allow the dimple to be located at any point on the specimen disc. However, unless there is a reason to do otherwise, the dimple should be positioned close to the center of the disc to obtain the maximum benefit of a supporting rim.

a) Place the specimen mount (and centering ring) on the turntable. The turntable and bottom of the mount may be wiped with lint-free paper, moistened with a small amount of vacuum grease, to remove any dirt and to ensure that the mount slides freely;
b) Gently position the light microscope on the housing surrounding the specimen turntable;

Note: The contacting surfaces of the microscope and housing must be clean or the microscope will not position correctly and the dimple will not be in the correct place.

c) Insert the connector into the illuminator power socket;
d) Rotate the microscope eyepiece to focus the center spot;
e) Focus the microscope on the specimen;
f) Slide the specimen mount horizontally until the desired point on the specimen coincides with the centering spot;
g) Check correct centering by turning on (Table) the turntable motor. The specimen should rotate about the centering spot. lf not, check the cleanliness of the contacting surfaces of the microscope and housing or center the microscope;
h) Remove the microscope.

STEP 5. CHECKING GRIDING WHEEL

a) Clean the dimple wheel bearing and a 15 mm spherical grinding wheel with a cotton swab and distilled water;
b) Install the spherical grinding wheel on wheel bearing and tighten screw;
c) Set a proper load (usually about 15~20gm) and grinding wheel speed. Check that both motors are off and lower the micrometer;
d) Place a steel mount, without any specimen, on the turntable and carefully, with the cam, lower the grinding wheel onto the specimen;
e) Raise the micrometer until the dial indicator has rotated just over one complete turn and the needle just reaches zero;
f) ZERO the micrometer digital display;
g) Lower the micrometer (turn clockwise) until its display shows ~100 um. The dial indicator initially displays the same reading;
h) Turn on the arm motor only. The dial indicator shows the diameter change of the grinding wheel. A well mounted good wheel should has vibration within 2 um. If the vibration is greater than 5 um, check the mount or cleanness of wheel bearing and wheel itself. If the problem is the wheel itself, change a new grinding wheel for best performance.

STEP 6-1. GRINGING BY SETTING THE DIMPLE DEPTH

a) Set a proper load (usually about 15~20gm) and grinding wheel speed. Check that both motors are off and lower the micrometer;
b) Place the mount, with specimen, on the turntable and center the specimen with microscope;
c) Carefully, with the cam, lower the grinding wheel onto the specimen;
d) Raise the micrometer until the dial indicator has rotated just over one complete turn and the needle just reaches zero;
e) Zero the micrometer digital display;
f) Lower the micrometer (turn clockwise until its display shows the required depth of dimple. The dial indicator initially displays the same reading;
g) With a cocktail stick, place a small amount of diamond compound (2-4 mn) on the wheel and on the specimen and moisten with small amount of distilled water. Don't let it run off the turntable;
h) Turn on both ARM and TABLE motors. The dial indicator shows the progress of dimpling. Set AUTO termination. When the dial indicator reaches within 2-3 um of zero, it will automatically switch off the specimen turntable and grinding wheel motors;
i) As the specimen nears the required thickness, it may improve the final specimen quality if the load and rotational speed of the grinding wheel are reduced;

Note: The specimen surface and grinding wheel must not be allowed to dry out and a small pocket of dilute paste should be present on the specimen at all times. It is recommended that the wheel and specimen are occasionally cleaned and fresh diamond compound applied.

STEP 6-2. GRINDING BY SETTING THE FINAL THICKNESS (an alternative step of STEP 6-1)

a) Set a proper load (usually about 15~20gm) and grinding wheel speed. Check that both motors are off and lower the micrometer;
b) Place a mount, without any specimen, on the turntable and carefully, with the cam, lower the grinding wheel onto the specimen mount;
c) Raise the micrometer until the dial indicator has rotated just over one complete turn and the needle just reaches zero;
d) Zero the micrometer digital display. This setting may be used for any number of specimens;
e) Raise the arm, remove the mount and attach the specimen. Replace the mount on the turntable. Center the specimen with the microscope;
f) Raise the micrometer (turn counter clockwise) until the display shows the required final thickness;
g) Carefully, with the cam, lower the arm onto the specimen - the dial indicator shows the thickness of material to be removed. This value plus the micrometer reading are the thickness of the specimen plus the thickness of the mounting wax;
h) With a cocktail stick, place a small amount of diamond compound (2-4 um) on the wheel and specimen then moisten with small amount of distilled water;
i) Turn on both motors and set AUTO termination. The dial indicator shows the progress of dimpling. When the dial indicator reaches within 2-3 um of zero, it will automatically switch off the specimen turntable and grinding wheel motors. As the specimen nears the required thickness, it may improve the final specimen quality if the load and rotational speed of the grinding wheel are reduced;

Note: The specimen surface and grinding wheel must not be allowed to dry out and a small pocket of dilute paste should be present on the specimen at all times. It is recommended that the wheel and specimen are occasionally cleaned and fresh diamond compound applied.

STEP 7. POLISHING

a) Take off the grinding wheel, clean with distilled water and save for future use;
b) Clean the wheel bearing and the polishing wheel;
c) Place polishing wheel and felt polishing ring on axle. Hold black shield of dimple wheel bearing and tighten screw to secure polishing wheel assembly;
d) Thoroughly clean off grinding compound with a cotton swab and distilled water;
e) Apply a small quantity of 1 um diamond compound (coarse polishing) to both the felt strip and specimen then moisten with small amount of distilled water;
f) Carefully lower the polishing wheel onto the specimen; lower the micrometer 300-500 um from the top of the specimen;
g) Turn on both motors and set autoterminator OFF. Let it run for 2-5 mins and stop.
h) Clean and run the above polishing steps again with 0.05 urn alumina suspension for fine polishing.

STEP 8. Power OFF and clean the wheels, felt rings, wheel bearing and turntable please!!

STEP 9. Specimen Removal

a) Slide the specimen mount off the turntable;
b) Submerge the mount and specimen in acetone with a piece of filer paper at bottom;
c) Rinse in distilled water for 5 min and dry in the air;

Key points:

1) Do not use excess glue for the grid;
2) Grinding finish thickness (specimen + grid) should be 70~100 um;
3) Avoid bubbles when mounting the specimen on the mount;
4) Remember to center specimen and check grinding wheel vibration prior to grinding;

G. GATAN DuoMill

1. Turn on the water supply behind the DuoMill. The two handles will face up when water is flowing.

2. Raise the main circuit breaker on the right side of the cabinet.

3. Start the roughing pump by depressing the upper face of "A" switch.

4. When pressure drops below 300 millitorr turn on the diffusion pump by depressing the upper face of "B" switch and allow chamber pressure to drop to 10^{-5} torr before proceeding.

5. Load specimen on the appropriate holder and screw the holder onto the top of the airlock piston.

6. Replace the specimen capsule.

7. Evacuate the airlock by depressing the "VAC" button intermittently until the backing pressure falls below 50 millitorr. Important: to avoid backstreaming of pump oils do not allow the backing pressure to exceed 300 millitorr.

8. Make sure the "RAISE" part of the "RAISE/LOWER" switch is depressed.

9. Open the argon cylinder valve fully. Open the delivery valve on the regulator. Delivery pressure should be about 10 PSI.

10. Depress the "LOWER" part of the "RAISE/LOWER" switch.

11. Once the specimen is in the work chamber depress the upper face of the "HIGH VOLTAGE" switch, set the process timer and turn on the argon solenoid valve switches located on the small - panel which also holds the argon metering valves. Do not disturb the "HIGH VOLTAGE" or "CURRENT LIMIT" knobs: they should be preset (black marks on panel should line up with the knobs' setscrew holes). Voltage should be about 5KV for the right hand guns and 4KV for the left. Gun current is limited to 1.3-1.5 milliamps, but this value won't show on the gauge.

12. Turn HV gun selector to the red position and adjust the red metering valve to give about .5-.6 milliamp of gun current. Turn HV gun selector to the white position and repeat. Note: the gun current will drift at first until the guns warm up and stabilize. Check gun current often. Finally turn the HV gun selector to the red and white position. Gun current should be 1.0-1.2 milliamps.
13. Fill dewar with LN2 if desired and position. Depress the upper face of the "SPECIMEN ROTATE" switch if desired.

14. To remove a specimen, depress the "RAISE" part of the "RAISE/LOWER" switch. Vent the airlock by depressing the "AIR" button. Remove the specimen capsule and unscrew the holder from the top of the airlock piston. If more thinning is needed screw the specimen holder back onto the top of the airlock piston and repeat steps 6, 7 & 10. Check the gun currents and adjust if necessary.

15. When finished thinning replace the specimen capsule, set the process timer to zero, depress the lower face of the "HIGH VOLTAGE" switch and turn off the argon solenoid valve switches.

16. Turn off the diffusion pump by depressing the lower face of "B" switch.

17. After about 30 minutes turn off the roughing pump by depressing the lower face of switch

18. Shut off the argon delivery valve on the regulator and argon cylinder valve.

19. Turn off the main circuit breaker on the right side of the cabinet.

20. Close the water valves.

H. EDAX Energy Dispersive X-ray Spectrometer (EDS) on the XL30-FEG

Sample preparation

Non-conductive samples must be coated sample with a thin carbon or metal film. Metal coating will interfere with some identifications. For precise quantitative work, the sample should be flat and polished. For semi-quantitative work other samples are acceptable.

Sample – detector geometry is an important parameter. The detector is above and at 45° to the sample. If the sample is not flat, it will be hard to get x-ray signal from the side of an irregular-shaped sample that is on the opposite side from the detector and slanting away from the detector.

Interaction volume

The detector collects data from a hemisphere-shaped region (the interaction volume) where the beam strikes the sample. This sphere can have a diameter of 0.5 -2 microns depending on the sample and accelerating voltage. If the object to be identified is smaller than this, there will be signal from the surrounding material.

X-ray types

Two types of x-rays come from the sample:

1. Characteristic x-rays - these are characteristic for each element, and are what is used to identify the element.
2. Continuous x-rays - these make the background along the bottom of the spectrum, and must be subtracted from the total for quantitative analysis.

Voltage setting

Each element has a critical energy for detection. This value can be found on the elemental chart on the wall behind the SEM, and it is also available in the EPIC table in the EDS software.

Set the SEM accelerating voltage at twice the critical energy of the elements of interest.

Accelerating voltage of 15 kV is good for elements up to Fe; for elements with atomic number higher than copper (Cu) use kV=20 or 30).

Instructions for using the EDS

1. Put sample in the chamber and start the SEM.

2. Select accelerating voltage according to your needs.

3. Set the WD (working distance) at 10 mm. Stage tilt should be at 0°.

4. Focus the electron beam on the spot you desire to analyze, or choose a selected area of desired size and shape.

5. On the EDAX PC start the GENESIS software.

6. Set acquisition parameters.
   - acquisition duration (preset time)
   - Amp time
   - verify that the microscope parameters match your SEM settings.

After you have focused the electron beam on the desired area of interest on the sample in the XL30-control program, set microscope and detector parameters, you may need to optimize spectrum acquisition by adjusting the microscope conditions (if possible) until the count rate in the CPS window is:

between 1000 and 3000 (for Amp time constant above 50 ms)
between 3000 and 10,000 (for Amp time constant below 50 ms).

X-ray signal intensity is controlled by the voltage selection, the spot size setting, and the aperture size. Change these parameters accordingly to get desired count rate.

7. Start spectrum acquisition. Wait until the preset time is reached or stop the acquisition when desired.
8. Save the newly acquired spectrum in .spc format.

9. Identify all the peaks in the spectrum. To ensure that you have identified all elements in a spectrum, you must identify all spectral peaks. Peaks can be identified automatically or manually. If peaks remain unidentified, they could be artifacts such as: Absorption, Escape or Sum peaks.

10. Spectrum quantification

There are two spectrum quantification tools:

- Background processing
- Full spectrum processing, including background subtraction.

After background correction is performed. The spectrum can be processed to extract quantitative or semi-quantitative data. By using one of the provided correction schemes. Use SEC method if no standards are available.

Use ZAF corrections for quantification with standards.

For proper quantification procedure consult the EDAX Genesis User's manual.

I. Lapping and Polishing Basics

1.0: Introduction

Lapping and polishing is a process by which material is precisely removed from a workpiece (or specimen) to produce a desired dimension, surface finish, or shape. The process of lapping and polishing materials has been applied to a wide range of materials and applications, ranging from metals, glasses, optics, semiconductors, and ceramics. Lapping and polishing techniques are beneficial due to the precision and control with which material can be removed. Surface finishes in the nanometer range can also be produced using these techniques, which makes lapping and polishing an attractive method for materials processing.

This paper describes some basics about lapping, including equipment setup, typical lapping techniques, and nomenclature.

2.0: Back to Basics

There are several techniques used for removing material from a particular workpiece (also called specimen in this discussion). Grinding, lapping, polishing, and CMP (chem.-mechanical polishing) are all techniques used for precise removal of material. A brief discussion of terms is needed to understand the basics of what is being referred to when these topics are discussed.

2.1: Grinding

Grinding can be defined as the rapid removal of material from a sample either to reduce it to a suitable size or to remove large irregularities from the surface. The grinding wheel or plate typically rotates at a high speed (around 200-1000rpm) and a coarse, bonded abrasive (> 40 μm) is used. Grinding is quick and relatively easy process but can cause deep subsurface damage in delicate materials. Typically grinding is applied to hard metals such as high carbon steels where rapid removal is essential and subsurface damage is not a critical parameter. For delicate materials the grinding process must be a balance of material removal and subsurface damage. In many cases it is advisable to

initially cut the specimen with a gentle mechanical method such as a wire saw. A properly prepared wire saw cut sample can eliminate the grinding process altogether.

2.2: Lapping

Lapping is the removal of material to produce a smooth, flat, unpolished surface. Lapping processes are used to produce dimensionally accurate specimens to high tolerances (generally less than 2.5 μm uniformity). The lapping plate will rotate at a low speed (<80 rpm) and a mid-range abrasive particle (5-20μm) is typically used. Lapping removes subsurface damage caused by sawing or grinding and produces the required thickness and flatness. Although the lapping process is less damaging than grinding, there are two regimes of lapping: free abrasive lapping and fixed abrasive lapping.

Free Abrasive Lapping is when abrasive slurry is applied directly to a lapping plate (e.g. cast iron). This is perhaps the most accurate method for producing specimens and causes the least amount of damage. Free abrasive lapping is accurate because of the rigid lapping surface which can be tailored to suit a particular material. Fixed Abrasive Lapping is when an abrasive particle in bonded to a substrate as with abrasive lapping films and SiC papers. Abrasive lapping films have various particles bonded to a thin, uniform polyester substrate and are also capable of producing a very flat surface. SiC papers are much thicker than the film and create the potential for rounded edges on the sample.

2.3: Polishing

Polishing is the removal of material to produce a scratch-free, specular surface using fine (<3μm) abrasive particles. Polishing is typically done at very low speeds using either polishing cloths, abrasive films, or specially designed lapping plates. Polishing with a cloth or lapping plate requires the use of free abrasive, and is a very low damage process when performed properly. Plate material and cloth material are critical when polishing a particular sample as the properties of these substrates are important in the final polish quality of the specimen.

Polishing with a lapping plate is a common process used in the case of metals and hard ceramic type materials. Polishing using copper composite plates or tin / lead lapping plates can produce high quality surface finishes with high removal rates. In many cases the use of a polishing cloth is required, and thus the selection of a proper polishing cloth is important. Polishing cloth properties needed depend on the application. If flatness is of primary concern, short nap cloths (such as Nylon) are used to maintain flatness. When the final surface finish is of primary concern, longer napped cloths (such as Rayon and Silk) are used. Many cloth materials today combine the best of both worlds, allowing flatness and surface finish combined to provide maximum performance. Polyurethane pads are commonly used for final polishing processes and produce excellent flatness and surface finish.

Polishing with abrasive films also produces excellent results. The flatness of the films combined with high removal rates makes them an attractive alternative to cloth and plate polishing methods.

2.4: Chem-mechanical Polishing (CMP)

Chem-mechanical polishing (CMP) is a technique that combines both chemical and mechanical polishing principles to achieve uniform removal rates of a highly composite specimen (such as integrated circuit device fabrication). CMP is

typically done using a hard polyurethane polishing pad combined with a slurry of finely dispersed alumina or silica particles in an alkaline solution. CMP combines the selectivity of chemical polishing with the mechanical removal properties of standard mechanical polishing techniques. The two combined give excellent selectivity and planarity and can be tailored to many different materials.

2.5: Abrasive Types

There is a wide selection of abrasives to choose from when selecting a lapping and polishing process. Selecting an abrasive is dependent upon the specimen hardness, desired surface finish, desired removal rate, lifetime, and price.
There are four basic types of abrasives that are used in lapping and polishing processes: silicon carbide (SiC), aluminum oxide or alumina (Al2O3), boron carbide (B4C), and diamond (C). All of these abrasives have distinct properties and are used for different materials and applications.

SiC: SiC is hard and generally has a needle or blocky structure. SiC is used in many applications where rough lapping is required. It seldom is used for polishing or applications that require smooth surface finishes.

Al2O3: Al2O3 is relatively hard and has a sharp, angular structure. Alumina is commonly used where fine surface finishes are required as it breaks down over time and gives excellent surfaces during lapping and polishing. Alumina is also relatively inexpensive.

B4C: B4C is harder than most other abrasives (excluding diamond) and has a blocky crystal structure. B4C provides excellent removal rates and is typically used when fast removal with moderate surface quality is needed.

Diamond: Diamond is the hardest material known and has a sharp, angular structure. Diamond is extremely useful in lapping and polishing due to its removal rates and surface finishing qualities. Diamond can produce excellent surface finishes combined with high removal rates.

MATERIAL	HARDNESS (KNOOP 100)	DENSITY	STRUCTURE
SILICON CARBIDE (SIC)	2450	3.22	BLOCKY, SOLID, SHARP
ALUMINA (AL_2O_3)	2000	3.97	BLOCKY, SOLID, ANGULAR
BORON CARBIDE (B_4C)	3000	2.51	BLOCKY, SOLID, SHARP
DIAMOND (C)	6000	3.51	SHARP, ANGULAR, SOLID

Table 1: Various Abrasive Materials and Associated Properties

3.0: Lapping and Polishing Equipment

Equipment used for lapping and polishing can vary from application. Typically what is required for lapping and polishing are the following:

1. A lapping and polishing machine with variable speed.
2. A polishing jig for holding specimens precisely.
3. Various lapping plates for different applications.
4. Workstations for controlling lapping fixtures and conditioning equipment.
5. Conditioning equipment for maintaining plate flatness.

There are various methods available to accomplish these tasks. South Bay Technology, Inc. has developed a series of equipment designed for this purpose and is described below.

3.1: Lapping and Polishing Machines

Lapping and polishing machines vary extensively depending upon the manufacturer. SBT has designed a set of instruments that are specifically designed for universal lapping and polishing applications. The Model 920 Lapping and Polishing Machine incorporates a precision spindle assembly housed in a solid cast aluminum casting to provide stable operation in any laboratory environment. Stability when lapping is critical in producing flat, precisely controlled tolerances on a given specimen. The motor is a high torque, variable speed motor that allows a wide range of speeds to be employed. Flexibility in speed control allows the instrument to be used as a grinding machine, high quality lapping machine, or polishing machine. During grinding high speeds are required, whereas lapping and polishing applications are generally completed at low speeds. The Model 920 also incorporates workstations, which allow for the use of precise Lapping and Polishing Fixtures. Each workstation has it's own speed control, allowing the user to precisely rotate the lapping fixtures or the conditioning ring. Lapping plates are held into place with three locating pins and are easily removable, allowing maximum flexibility in processing specimens. Various materials can be selected for the lapping plates, ranging from aluminum, cast iron, and glass.

Below is an image showing the Model 920 with a typical setup for lapping.

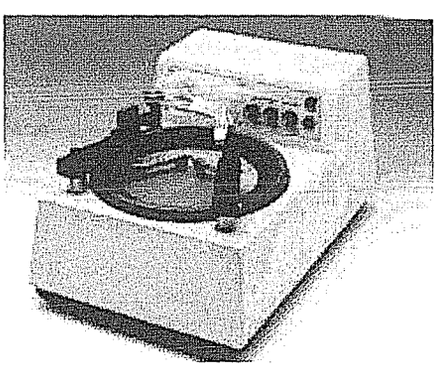


Figure 1: Model 920 Lapping and Polishing Machine for precise lapping and polishing applications.

3.2: Lapping and Polishing Fixtures

Lapping and Polishing fixtures are perhaps the most critical part of any lapping and polishing system. The lapping fixture is what holds the specimen during processing and ultimately will determine the final specimen quality. South Bay Technology has been a leader in the manufacture of precision lapping fixtures for any application. In general, the fixture typically performs like a piston, with a housing used for support of a dynamically sliding piston assembly. Specimens are attached to the base of the piston using vacuum mounting or wax mounting techniques. The piston movement is controlled with a precision dial micrometer at the top of the fixture, and thus allows the precise removal of material in various increments determined by the user.

The base of the housing contains a wear resistant ring that supports the fixture, controls the flatness and parallelism of the specimen, and gives stability to the fixture.

Below is a basic image of a typical lapping and polishing fixture.

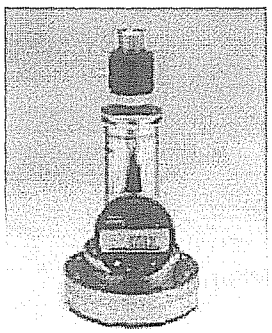


Figure 2: Model 164 Lapping and Polishing Fixture used for precision lapping and polishing of wafers and other materials.

3.3: Lapping and Polishing Plates

Lapping and polishing processes are performed on a hard, metal plate used in conjunction with abrasive suspensions such as diamond, silicon carbide (SiC), aluminum oxide (Al2O3), or boron carbide (B4C). The metal lapping plate selected depends upon the desired material removal rate, the surface finish desired, the hardness of the specimen being lapped, and the flatness requirement.

Plate selection can play a critical role in the production of high quality specimens. Lapping plates can be flat or grooved depending upon the desired application. Grooved plates provide greater removal rates and prevent the abrasive from squeezing out from between the plate and the specimen.

Cast Iron (Fe): Cast iron lapping plates are used for rough lapping and stock removal of materials. Specimens around 8-10 on the Mohs Hardness Scale can be lapped using cast iron plates. Cast iron produces a gray surface finish and provides high removal rates.

Composites: Composite plates are used for rough lapping and stock removal of materials. Specimens of 7-10 on Mohs Hardness Scale can be lapped on composite plates. These plates produce medium quality surface finishes with very high removal rates.

Copper (Cu): Copper plates are used for both rough and fine lapping of materials. Specimens around 9-5 on the Mohs Hardness Scale can be lapped using copper plates. They provide high quality surface finishes with intermediate removal rates.

Tin/Lead (Sn/Pb): Tin lead plates are used for fine lapping and polishing of materials. Specimens around 2-4 on Mohs Hardness Scale are lapped with tin lead plates. They provide high quality surface finishes with low removal rates.with low removal rates.

Polishing can also be carried out using a polishing cloth attached to a substrate plate (either glass or aluminum typically) using aluminum oxide, colloidal silica, or diamond suspensions. Use of polishing cloths can be advantageous due to the high quality surface finish they produce. However, it must be noted that for extended polishing times the use of a cloth will sometimes lead to wavy surfaces and edge turning, which can be detrimental to the specimen quality. Proper selection of polishing cloths and process times is key in the successful use of a cloth for final polishing of specimens.

3.4: Plate Conditioning

Plate conditioning is a process by which the lapping plate surface is machined to maintain the flatness of the lapping plate and to "pre-condition" the lapping plate surface with the abrasive being used for the lapping process.
Plate conditioning is important for lapping applications where flatness and parallelism of the specimen is critical. Specimen quality is a direct result of plate condition, and therefore proper maintenance of the lapping plate is crucial in preparing high quality specimens.

Conditioning of the lapping plate is affected by two primary parameters: a) position of the conditioning ring, and b)weight of the conditioning ring. The conditioning ring is generally a cast iron or stainless steel ring used with the abrasive desired or with diamond plated to the bottom of the ring. Conditioning of the lapping plate also helps maintain high removal rates during long lapping operations.

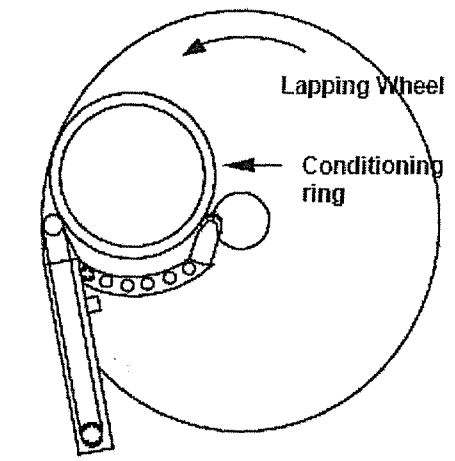


Figure 3: Schematic illustration of the conditioning process used on the Model 920. A workstation (Model 92002) is set up on the Model 920 and a conditioning ring is positioned on the lapping plate surface. The conditioning ring is used with abrasive slurry to make the plate flat.

Model 92002

3.4.1: Conditioning Basics

Proper positioning of the conditioning ring can help bring a plate back into flatness if the plate has become 'out of flat'. Often times the plate will resemble a wavy shape due to the positioning of lapping and polishing fixture inside the radius of the lapping plate. If the lapping plate has become concave in shape, then the conditioning ring should be adjusted to the outer diameter of the plate. The extra work done on the lapping plate will then bring the plate
back into flatness in a short time, provided the plate is not severely out of flat by more than 0.003" (75 µm). If the plate shape is convex, the conditioning ring should be positioned on the inner portion of the plate. Below are examples of this process.

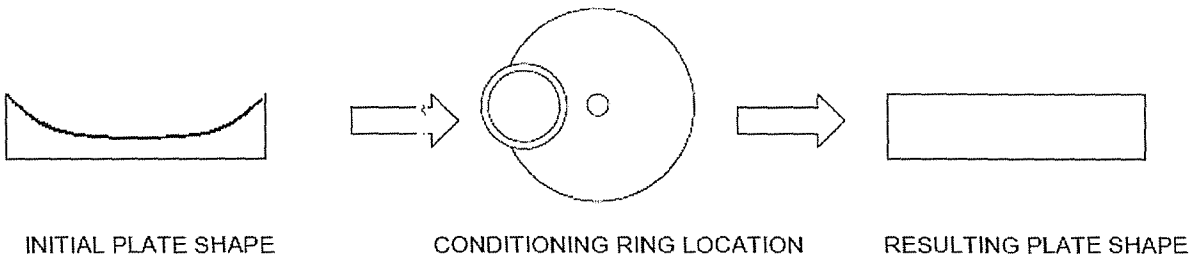


Figure 4: Schematic illustration of the conditioning process. The initial plate shape is a concave shape. Positioning the conditioning ring on the outer diameter of the plate will cause the highest point of the plate to be lapped faster than the inner portion, creating a flat plate surface.

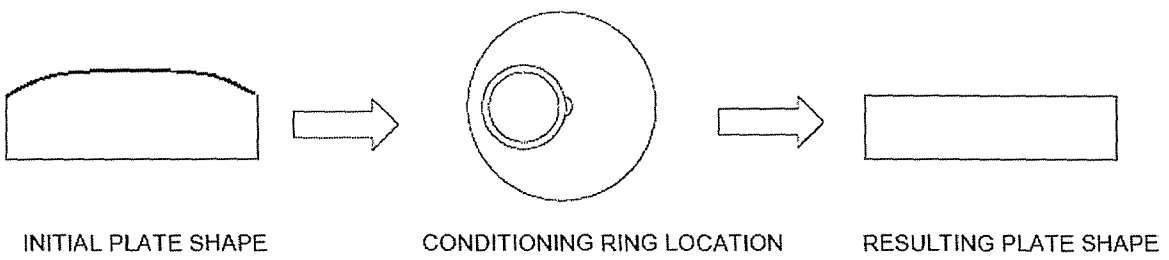


Figure 5: Schematic illustration of the conditioning process for the convex plate case. Positioning the conditioning ring to the inner portion of the plate will cause the high spot in the center to be lapped faster than the outer diameter, creating a flat plate surface.

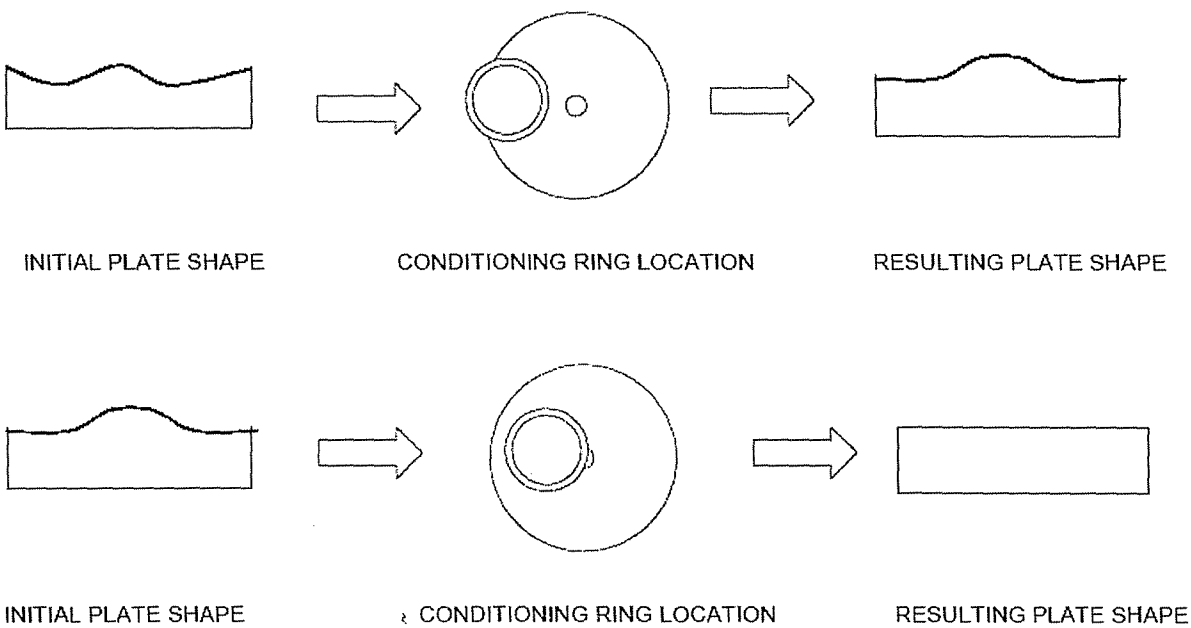


Figure 6: Schematic illustration showing a two step conditioning process. The top figure shows a plate with a wavy surface due to a small lapping fixture being used without a conditioning ring. The initial conditioning is completed with the conditioning ring on the outer diameter of the plate. Once the outer

portion of the plate has been lapped down, the conditioning ring is moved to the center of the plate to lap it flat.

4.0: Conclusion

The line of SBT precision lapping and polishing equipment can be used for a wide range of lapping and polishing applications where precise, high quality specimens are required. Use of the Model 920 Lapping Machine combined with the SBT series of Precision Lapping and Polishing Fixtures enables the user to closely control the lapping process and create excellent specimens.

From MEMS wafer planarization to the edge polishing of electro-optical waveguides, SBT has a complete solution to fit any applications need.

J. GATAN PIPS Model 691

Please Note:
The purpose of this document is to remind you of the essential points for the safe operation of this instrument. It is NOT intended as a substitute for proper training.

START-UP PROCEDURE

- Open main valve on Argon gas cylinder (DO NOT adjust other valves).
- Check Argon supply pressure = 25 psi.
- Switch on the ion mill using the POWER switch on the front panel.
- Diaphragm and molecular drag pump will start and digital display will light up.
- Red HIGH DP indicator will illuminate until backing pressure <12 Torr.
- After ~15 mins, Green MDP indicator will light up — MDP at 70% running speed.
- Chamber pressure will now register on vacuum gauge.
- Check that upper part of AIRLOCK CONTROL switch is depressed (airlock is up).
- Check that airlock cover is in place.
- Wait until PENN IN G GAUGE is < 4x10 "5 Torr before proceeding.
- Perform Ion-Gun Purge and Gas-Flow Adjustment as described in sections 3.5 and 3.6 of the GATAN Instruction Manual for the PIPS.

SAMPLE LOADING

- Mount sample into sample holder (These holders are very expensive- be careful!)
  - Use mounting block for clamp-type holder.
  - Use mounting wax and hot plate for post-type and glue—type holders.
  - Mount cross—section sample with interface parallel to long axis of holder.
- Confirm that upper part of AIRLOCK CONTROL switch is depressed (airlock is up).
- Vent airlock chamber by holding down the VENT button.
- Remove airlock cover (keep it clean!)
- Use special tweezers to load clamp-type sample holder into airlock.
- Ensure long axis of holder is parallel with front panel of ion mill.
- Ensure sample is properly seated, but do NOT push down hard!!
- Clean glass window if required.

- Replace airlock cover.
- Depress VAC button while gently rotating airlock cover to properly seat o-ring.
- Once airlock chamber reaches a safe vacuum level, green VAC light will illuminate.
- Depress lower part of AIRLOCK CONTROL switch to lower sample into chamber.

ION MILLING

- Rotate ion guns to desired angle (note TOP marking on each gun).
- Switch on ION GUN GAS FLOW CONTROL switches – green LED will light.
- Set sample ROTATION SPEED to desired speed (0-6 rpm).
- Set required milling time using up/down arrows on HIGH VOLTAGE TIMER (clock scale is MIN:SEC).
- Set the ION GUN control to desired energy.
- Set ION BEAM MODULATOR:
  - OFF: Ion beam always on. For single-side milling ONLY. Both guns TOP ONLY. Use post-type holders ONLY.
  - DOUBLE: Ion beam on in 2 sectors of 60° each (both guns).
  - SINGLE: Ion beam on in 1 sector of 60° (both guns).

NOTE

When using clamp-type holder: Set MODULATOR to DOUBLE or SINGLE.

Operate PIPS with MODULATOR set to OFF ONLY with post-type holder:

- Press START. Shutter will close below window- check that this occurs!

SAMPLE EXAMINATION

- Swing optical microscope into position over airlock
- Switch on light source (6 maximum).
- Switch on monitor.
- Open shutter using switch on top right-hand side of airlock.
- Find image.
- Optical microscope should be aligned- you only need to adjust the focusing.
- Close shutter and turn off both light source and monitor when finished.

SAMPLE REMOVAL

- Check that milling has finished- press STOP if not.
- Switch off gas flow to ion guns (GAS FLOW CONTROL switches).
- Depress upper part of AIRLOCK CONTROL switch to raise sample into airlock.
- Press and hold the VENT button until cover is loose.
- Remove sample holder with special tweezers, remove your sample and store sample holder in box.

- RETURN SAMPLE HOLDERS TO TEM SPECIALIST.
- Replace cover and evacuate airlock by pressing VAC and gently rotating cover
- Check to see if anyone is using the ion mill after you
- You MUST shut down the ion mill if there is no-one using it after you!

SHUTDOWN

- Ensure that the airlock is in raised position and ion mill has been properly evacuated.
- Check that GAS FLOW CONTROLS to both guns are off.

- Turn off the ion mill using the POWER switch On the front panel.
- Close the main valve on the Argon cylinder.

K. RMC ULTRAMICROTOME MT-X

Mastering the MT-X is easy, especially if you take the time to read through the following description.

The MT-X power switch is located on the back of the control unit. The red LED displays and the LED lamps on the control unit will illuminate when the instrument is turned on. The red lamp underneath the RESET button will also illuminate. The advance mechanism automatically resets to the beginning of travel when the instrument is turned on, emitting a faint motor noise. Refer to Figures 1, 2, and 3 while reading section II to help in acquainting yourself with the instrument.

A. The Main Unit

A.1 HANDWHEEL.

Each rotation of the handwheel causes the cutting arm to go through a complete stroke, up and down. The up stroke corresponds to the retract half of the cutting stroke. The cutting arm visibly retracts by a minimum of 80 um during this part of the stroke. If the handwheel is left in any part of the up stroke, it will beep two short beeps every 10 seconds to remind you that the arm is in a retracted position and you should not advance the knife to the specimen. In the down, or cutting half of the stroke, the arm extends outward for cutting.

During rotation of the handwheel there are two click sounds, one at the top of the cutting stroke and one at the bottom. These are the normal sounds made by the retract mechanism. The ample retract of your MT-X assures that no water will be picked up from the trough by the specimen during the up stroke, even when cutting thick sections from large block faces.

A. 2 FIBER OPTICS LIGHT PORT.

Both of the fiber optics pipes from your accessory box plug into this port (see figure 3). The thick pipe plugs into the back of the upper stage for backlighting of your knife and specimen during knife approaches. It may also be hand-held to intensely spot—illuminate your specimen, such as when trimming on the trimming post.

The thinner fiber optics light pipe plugs into the side of the transilluminated specimen holders. It illuminates the specimen from behind the specimen block.

The lower Lighting System button on the control unit is used to turn the fiber optic lamp on and off

A.3 UPPER STAGE.

The upper stage slides directly on to the top of the lower stage and is clamped firmly into position by the black Clamping Knob on the right side of the lower stage (see figure 3). When this knob is released, the upper stage can slide from

front to back. This feature can be used to bring the knife to within 1 or 2 mm from the specimen before beginning the final approach.

The upper stage will also rotate 360° while attached to the lower stage. This allows the user to adjust the stage to any position and to view the back side of the diamond knife.
There are two Stage Rotation Knobs, located on either side of the upper stage. These permit precise rotational alignment of the knife to the specimen.

The small knob on the left side of the upper stage is the Clearance Angle Adjustment Knob. To set this angle, first remove the fiber optics light pipe from the knife holder, and then loosen the small Knife Clamping Knob on the right side of the upper stage which secures the Knife Holder in place. Next, set the Clearance Angle Adjustment Knob to the desired angle. For most glass and diamond knives, 4° is a good angle to try first. Your diamond knife may specify a different angle; usually this angle is written on the knife storage box.
Next, tighten the knife clamping knob. Finally, replace the fiber optics light pipe into the knife bolder.

A.4 LOWER STAGE.

The lower stage incorporates a system of special roller bearings for maximum stability. It can be shifted to the left or right using the lateral knob on the right-hand side. Precision forward and backward movements are made with the Coarse and Fine Advance Knobs. When turning these knobs, you can feel the smooth drive mechanism, which allows you to quickly and precisely advance the knife to the specimen. Rotating either of the knobs counterclockwise advances the knife towards the specimen. Likewise, rotating the knobs clockwise causes the knife to move away from the specimen.

The Coarse Advance Knob is the larger of the two knobs. Graduations on this knob are in micrometers (µm or microns). Each short mark represents 5µm of travel. One complete rotation advances the stage 500 µm, or 0.5 mm.

The Fine Advance Knob is smaller and is located furthest to the left. This knob is for very precise approaches of your knife to the specimen. It can also be used to rough face your block by advancing a small increment for each cut. This knob is graduated with a vernier scale (see Figure 4).

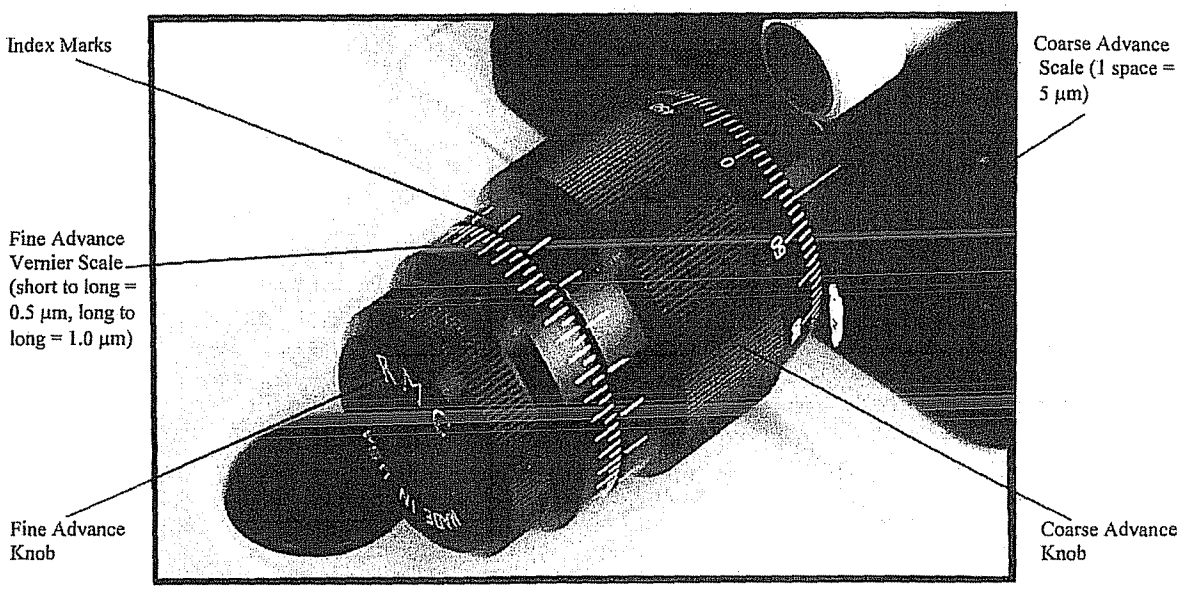

Index Marks
Coarse Advance Scale (1 space = 5 µm)
Fine Advance Vernier Scale (short to long = 0.5 µm, long to long = 1.0 µm)
Fine Advance Knob
Coarse Advance Knob

figure 4. **Coarse and Fine Advance Knobs**

There are index marks around the coarse advance knob which face the graduations on the fine knob. Rotate the fine knob until one of the longer marks lines up with one of the opposing marks on the coarse knob. Now, if you rotate the knob counterclockwise just a small amount until the next short mark lines up with the same index mark on the coarse knob, the stage will have advanced 0.5µm. Rotating further to the next long mark advances another 0.5 µm. Turning the knob from where one long mark lines up with an index mark to where the next long mark lines up with the same index mark advances 1.0 µm. Index marks are provided at intervals all around the coarse knob so that one small or large mark will always be lined up with an index mark in any position.

To prove to yourself how these scales work, watch how turning the fine knob through 5 long marks causes the scale on the coarse knob to indicate 5nm of travel.

A.5 ARC SEGMENT MOUNT.

The arc segment mount is stored in your accessory box. To use the arc segment mount, insert the mounting pin into the end of the cutting arm (see figure 3). Normally you may want the arc segment mount oriented so that the Specimen Rotation Knob is on the right side, and the Arc Adjustment Knob is on the left side, with the arc positioned vertically. To secure the arc segment mount to the cutting arm, tighten the Locking Screw.

For special purposes, you may want the arc oriented horizontally, such as to cut your specimen block at a different angle. You may orient the sample at angles up to 45° by tightly screwing another mounting pin (P.N. 75155) into the back and inserting this pin into the cutting arm.

Your Specimen Holder will fit into the arc segment mount. If the specimen holder has a 10 mm mounting pin, it may be inserted directly into the arc segment mount. If not, an Adapter (P.N. 75363) must be screwed tightly onto the thread. To mount a specimen holder, insert it into the arc segment mount and rotate the specimen rotation knob until you can see a set screw through the specimen locking screw hole. Firmly tighten this with the wrench provided in your accessory box.

Now try out the specimen rotation knob and the arc adjustment knob to see how these adjustments allow you to rapidly adjust your specimen to the desired orientation for sectioning.

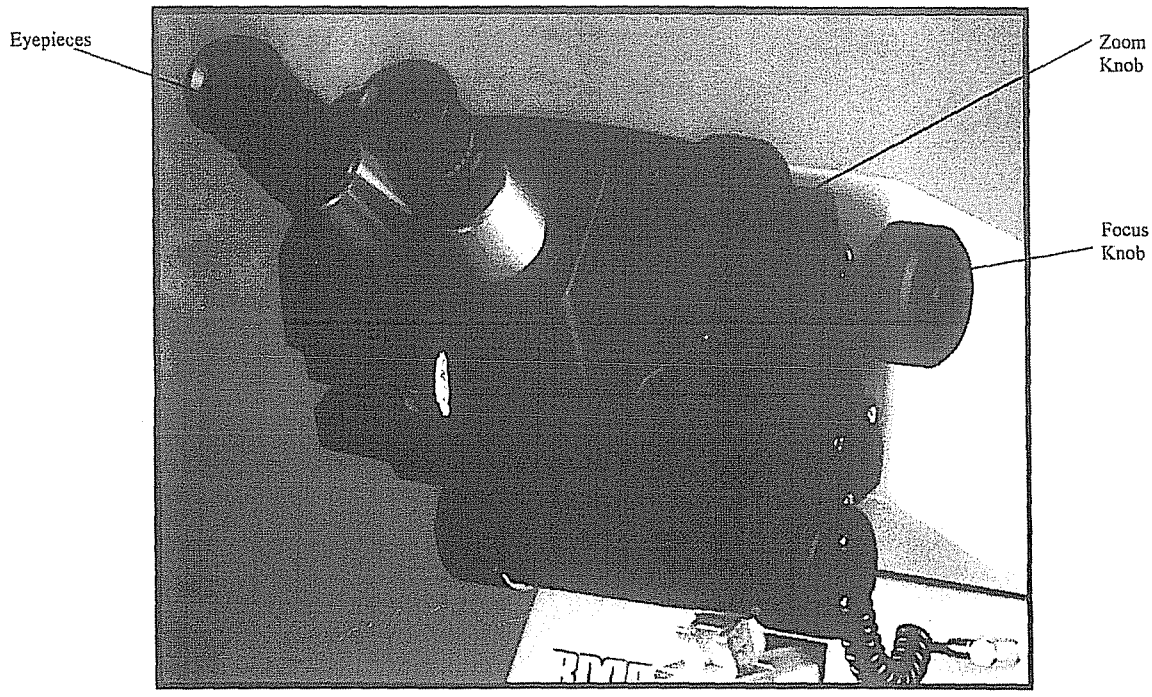


figure 5. **Stereomicroscope**

A.6 STEREOMICROSCOPE HOLDER.

This holder permits a complete range of movement of your stereomicroscope for optimum viewing (see figure 5). The stereomicroscope can be swung to the left and right, but be sure you do not go so far as to stretch the coiled black cord which goes to the overhead light. The stereomicroscope may also be tilted up and down. This movement allows you to set the optimum viewing angle for approaching your knife to the specimen and to select the best angle for viewing the sections as they float out on the water during sectioning.

The eyepieces are adjusted by moving them together or apart to match the distance between your eyes. When first looking through the stereomicroscope, you may not recognize what you see if it is set at too high a magnification or is out of focus. To set the lowest magnification, rotate the Zoom Knobs downwards. Now rotate the Focus Knobs up and down to focus. To position the knife edge or specimen in the center of view, you can rotate the Forward Travel Knob (see figure 1) to move the stereomicroscope towards or away from you. Coarse positioning of the stereomicroscope from left to right is best done by simply grasping it and pulling it right or left until it is approximately centered over the knife and specimen. Precise, fine centering is easily done by turning the small Lateral Adjustment Knob on the left underside of the stereomicroscope holder.

A.7 OVERHEAD DIFFUSE LIGHT.

The overhead diffuse light is attached to the stereomicroscope holder. It is fully adjustable. The light may be tilted to optimize illumination. To do this, loosen the small black Lamp Tilt Locking Knob located on the left side of the stereomicroscope focusing block. The overhead diffuse lamp can also be rotated to further optimize illumination. Simply loosen the small black Lamp Rotation Locking Knob on the top of the round lamp housing.

B. The Control Unit

B.1 SECTIONING

1.a Cut. This switch controls automatic cutting under motor power. To start or stop automatic cutting, press the button once. The display above the cut button has 2 LED lamps. When the bottom green lamp is lit, the cutting arm is in the cutting window of the cutting stroke (see below). When the top yellow lamp is lit, the cutting arm is outside of the cutting window. Even when the motor is not running, these lamps remain lit to indicate whether the cutting arm is inside or outside of the window.

1.b Foot Pedal. The foot pedal serves the same function as the cut button on the control unit. The foot pedal receptacle is located on the back panel of the control unit, below and to the left of the on/off switch. To insert the foot pedal switch into the receptacle, line up the red dots and insert.

1.c Cutting Window. These two buttons are for setting the start and end positions of the cutting zone. Depressing these buttons while the instrument is in automatic cutting mode will have no effect. The automatic cutting motor must be off to use these buttons. To set a cutting window, turn the handwheel until the specimen is about 1 mm above the knife edge, then depress the top cutting window button to set the upper limit of the cutting window. The beeper will sound twice (Beep-Beep) if the position has been accepted. Then, turn the handwheel until the specimen block face just passes the knife edge. Now depress the bottom cutting window button to set the lower limit of the cutting window. Again, the beeper should sound twice. The cutting window is now set. The computer will not allow the user to set a cutting window with the upper position below the lower position or vice versa. If this is attempted, the computer will beep once to notify the user that the position has not been not accepted. The beeper will sound only once if either of the buttons is depressed when the cutting arm is outside of the cutting portion of the cutting stroke. This is to warn you that this is not a permissible position and it has not been accepted by the computer.

Once the cutting window has been set, the cutting arm will slow down to the speed displayed on the cutting speed display as it passes through the cutting window.

1.d Cutting Speed. This button sets the cutting speed in mm/sec. To change the speed, depress the + or - button until the desired cutting speed is displayed. For most specimens embedded in epoxy plastics a speed between 0.5 and 1.5 mm/sec will give the best results. This speed is in effect only when the arm is passing through the cutting window described above. When the cutting arm passes out of this cutting zone, it retracts at the retract speed.

1.e Section Thickness. The section thickness in nanometers is indicated in this red display. The thickness may be changed by depressing either the + or the − button. One nanometer incremental changes are made by depressing either button once. If you hold the button down longer, it will begin to steadily change the value. Alter 10 seconds the value will change at a rapid rate. Remember that 1000 nm is equivalent to 1.0 µm (1.0 micrometer or micron).

1.f Memory Storage. There are 5 buttons in this area. The buttons with the numbers 1 to 4 above them serve to activate any of the 4 stored sectioning thickness programs. These allow you to store section thicknesses which you commonly use. To create a program, depress one of the buttons. The green lamp above the button will light to indicate that the program has been selected. Then set the desired section thickness. Next, depress the memory storage button. The section thickness is now stored in the designated program. To recall the section thickness in any program, simply depress the corresponding button. If the section thickness stored is greater than 250 nm, the display flashes; if the button is not pressed again within seconds to confirm, then the system defaults to the previously selected setting. This feature prevents the operator from accidentally selecting a thick section which might damage the knife edge. It is helpful to set up separate programs for commonly used section thicknesses for ultrathin and thick sectioning. The section thicknesses can always be quickly adjusted at any time, and stored with the memory storage button or recalled by depressing one of the program buttons.

B.2 LIGHTING SYSTEM

2.a Overhead Diffuse Light. The overhead diffuse light is turned on and off using the top lighting system button on the control unit. The top yellow lamp on the control unit will be lit to indicate that this light is turned on.

2.b Fiber Optic. The fiber optics light is turned on and off using the bottom lighting system button on the control unit. The bottom yellow lamp will be lit to indicate that this light is turned on.

B.3 STEP ADVANCE

3.a Left Button. This button advances the cutting arm towards the specimen by the amount shown in the section thickness display. This is very useful for making final approaches of the specimen to the knife.

3.b Right Button. This button moves the cutting arm away from the specimen by the amount shown in the section thickness display. This is useful for backing the specimen away from the knife edge if you wish to discontinue sectioning for a moment.

3.c Reset. This button will reset the advance mechanism to the beginning of travel. When the arm is reset, there will be 200 µm of available advance. The small red lamp below the reset button will remain lit until the advance mechanism is fully reset. This should take less than 20 seconds. No button commands will be recognized until the reset is completed.

L. South Bay Tech. Disk cutter 360

3.0: Instrument Operation

3.1: Specimen Mounting There are two stage options for mounting specimens with the Model 360, One option is to use the 1" or 2" work tables with aluminum and graphite plates (see figure 2A). And the second is utilizing the circular mounting plate with a magnetic slurry ring (see figure 2B). The instructions below show the basic method for mounting a specimen using either of the listed mounting stages.

Procedure for Option #1

1. Place the aluminum mounting plate (4) onto a hot plate and heat to 135° C.
2. Apply enough wax (MWH 135) to the aluminum block top surface so it will hold the graphite mounting plate (2),
3. Affix the graphite plate on top of aluminum plate.
4. Apply wax to the graphite plate top surface.
5. Place the specimen onto the mounting plate and remove from the hot plate.
6. Gently press the specimen down into the wax to create a thin; uniform layer of wax.

SPECIAL NOTE: This mounting option disables the functioning of the automatic shutoff system. In order for the automatic shutoff to operate specimen mounting option #2 must be used.

7. Once the specimen has cooled, place it onto the worktable and tighten the set screw (7) to lock the specimen into place onto the worktable.
8. Place the worktable into the worktable support (5) and lock it into place using the set screw (3).
9. Place the entire assembly onto the magnetic plate (8).
10. Place the magnetic plate onto the Model 360 mounting base and align with the three guide pins on the mounting base. The beveled edge of the mounting plate should be positioned into the corner of the three guide pins. (See Figure 2A arrow, Top View below).

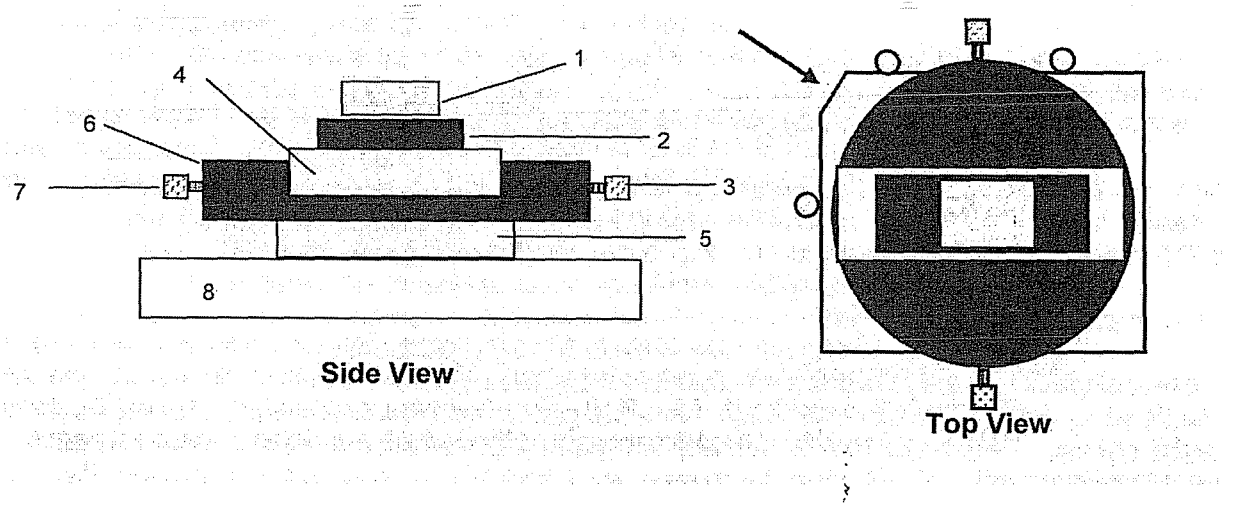


Figure 2A: Illustration of option #1 setup used for specimen mounting. 1- Specimen; 2- Graphite mounting plate; 3- Set screw; 4- Aluminum mounting plate; 5- Work table support; 6- Work table; 7- Set screw; 8- Magnetic plate.

MODEL BSO INSTRUCTION MANUAL

Procedure for Option #2

1. Place the circular specimen mounting plate (3) onto a hot plate and heat to 135° C.

2. Apply a small amount of wax (MWH 135) to the top surface of the mounting plate.
3. Place the specimen onto the mounting plate and remove from the hot plate.
4. Gently press the specimen down into the wax to create a thin, uniform layer of wax.
5. Once the specimen has cooled, place it onto the magnetic plate (4).
6. Place the magnetic slurry ring (2) over the specimen and specimen mounting plate
7. Place the entire assembly onto the Model 360 mounting base (5) and align with the three guide pins on the mounting base. The beveled edge of the mounting plate should be positioned into the corner of the three guide pins.

(See Figure 2A arrow, Top View below).

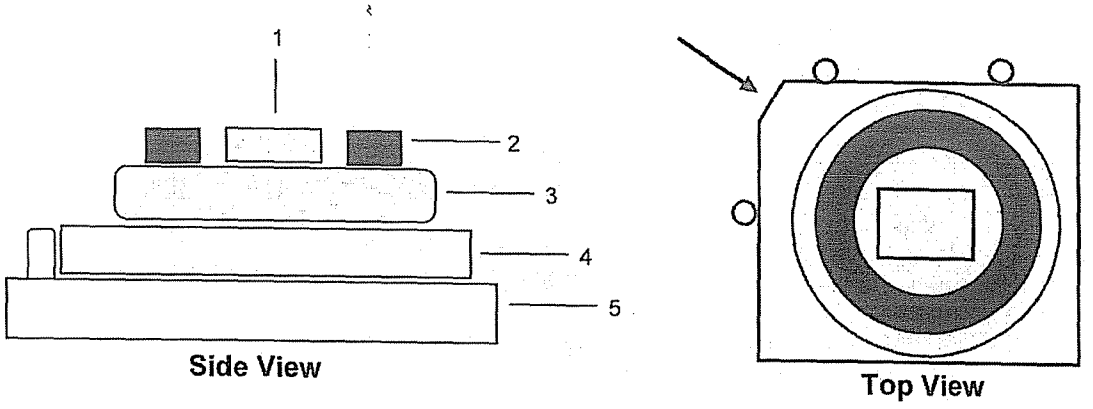


Figure 2B: Option #2 illustration of the setup used for specimen mounting. 1- Specimen; 2- Magnetic slurry ring; 3- Specimen mounting plate (circular);4- Magnetic plate; 5- Magnetic base.

3.1.1: Specimen Alignment

At certain times it may be necessary to cut small discs of pre-defined areas on the specimen. This requires alignment of the area of interest with the cutting tool. There are a couple of methods which can be used to accomplish this task, which vary with the geometry (or size) of the area to be cut and I the frequency with which this procedure will be used.

Standard Alignment

For bulk cutting or cutting easy to see structures, the specimen is aligned by moving the mounting block or plate relative to the cutting tool. This procedure is very quick and simple and is used for aligning bulk specimens for cutting. Finer alignment procedures should be done using the Template method or the Alignment Microscope method which are detail in the next pages.

Template Alignment

Another method often employed for selecting specific areas for cutting on the Model 360 is the use of a template. This procedure involves cutting a hole in a material (such as cardboard) the size of the cutting tool to be used. The material is then placed over the area of interest prior to cutting and the tool is then aligned to the hole in the template material. This is a quick and easy method for aligning the cutting area as defined by the tool. Below is a description of the procedure used for using a template for specimen alignment.

1. Obtain a small square piece of cardboard, silicon, or any material desired for making a permanent template.

2. Cut out a small hole the size of the desired cutting tool to be used for making the disc shaped specimen.
3. Mount a specimen as described in Section 3.1.
4. Place the template over the area desired for cutting.
5. Align the specimen such that the template is aligned with the cutting tool and the cutting tool will cut the specimen in the exact area of the template.
6. Remove the template and cut the specimen.

Model 36001 Alignment Microscope (optional accessory)

The Model 36001 Alignment Microscope was designed to facilitate the precision alignment of small features on a specimen for disc cutting. Listed below is the basic method used for aligning a specimen.

1. Mount the desired cutting tool into the Model 360.
2. Place the magnetic plate into the Model 360 as normal.
3. Cut a small groove into the magnetic plate to mark the position of the cutting tool relative to the magnetic plate location.
4. Remove the magnetic plate from the Model 360 and place it onto the microscope stage. Be sure that the orientation of the magnetic plate is identical to that of the Model 360.
5. Remove the protective plastic cover of the alignment microscope by turning the cover counter clockwise.
6. Turn the focus knob of the microscope (2) clockwise until it reaches the lower limit of its range. This will create the shortest working distance from the magnetic plate (7) and the objective (5).
7. Lower or raise the head assembly of the microscope by loosening the locking nut (3) on the side of the microscope until the groove in the magnetic plate becomes focused in the eyepiece. Lock the microscope into place by tightening the locking nut.
8. Adjust the base plate (B) of the microscope to center the groove in the magnetic plate. This is done by loosening the three set screws which attach the plate to the base of the microscope. Position the plate by slightly moving the plate until the groove is exactly centered in the objective eyepiece.
9. Once the microscope has been aligned to the cutting tool groove, do not change its position.
10. Turn the focus knob of the microscope (2) counter—clockwise until it reaches the upper limit of it's range. This will create the longest working distance from the magnetic plate (7) and the objective (5), allowing the largest possible specimen configuration.
11. Place the entire specimen holder assembly (Figure 2, Top View) onto the magnetic mounting plate (7). Be sure that the plate does not move from its position.
12. Turn the focus knob of the microscope (2) clockwise until the specimen is fully focused.
13. Locate the area of interest by moving the entire specimen holder assembly or by loosening the set screw in the work table and adjusting the position of the aluminum mounting block. Both adjustments may be necessary to locate the area of interest.

NOTE: Be sure not to move the position of the magnetic mounting plate.

14. Locate the desired area of interest and center it in the objective eyepiece. This will be the area which is cut by the Model 360.
15. Remove the specimen holder assembly and the magnetic mounting plate and place the entire assembly onto the Model 360 stage in the same orientation as setup on the microscope.

NOTE: Be sure that the position of the specimen holder assembly and the magnetic plate are not changed. Any change in position will cause a misalignment of the specimen to the cutting tool.

16. Cut the specimen.

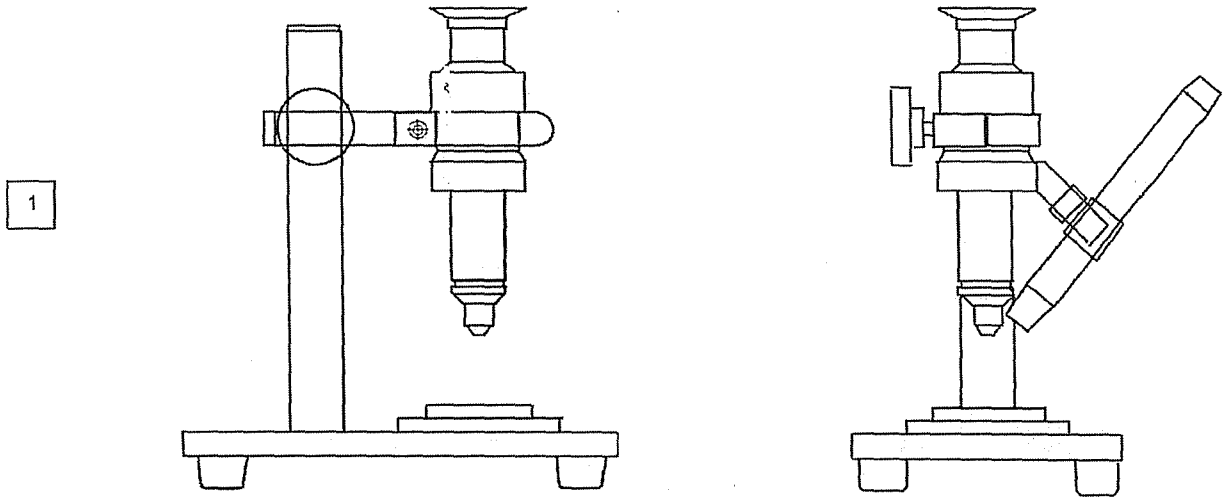


Figure 3: Illustration of the Model 36001 Alignment Microscope. 1- Objective eyepiece; 2- Focus knob; 3- Vertical positioning set screw; 4- Light; 5- Objective; 6- Specimen post; 7- Magnetic mounting plate; 8- Positioning plate.

3.2: Cutting Tool Installation

Prior to cutting a specimen the cutting tool must be mounted into the Model 360. Proper placement and mounting of the tool is critical in producing reasonably damage free specimens which do not have severe edge chipping and flashing. Tool installation is a very simple process and is described below in detail for convenience.

1. Loosen the drill assembly by loosening the collet nut (6). This is done by placing the smaller wrench on the shaft bolt (2) and the larger wrench on the collet nut (6). Turn the collet nut counter-clockwise until the whole assembly becomes loose.
2. Remove the chuck collet (5) and the tool collet (4).
3. Place the desired cutting tool (7) into the collet nut and then place the tool collet into the chuck collet. The cutting tool should fit snugly into the collet and the collet should rest in the chuck collet.
4. Lock the entire assembly in place by pressing it into the shaft of the Model 360 until the collet nut (6) rests against the threads (3) of the shaft.
5. Lower the entire shaft of the Model 360 until the cutting tool comes into contact with the base plate of the unit.
6. Gently press down onto the handle until the tool slides all the way into the chuck collet assembly. This ensures that the tool will be centered and the cutting tool will be as stable as possible.
7. Tighten the entire chuck collet assembly by holding the shaft bolt (2) in place and turning the collet nut clockwise.
8. The unit is now ready to cut a specimen.

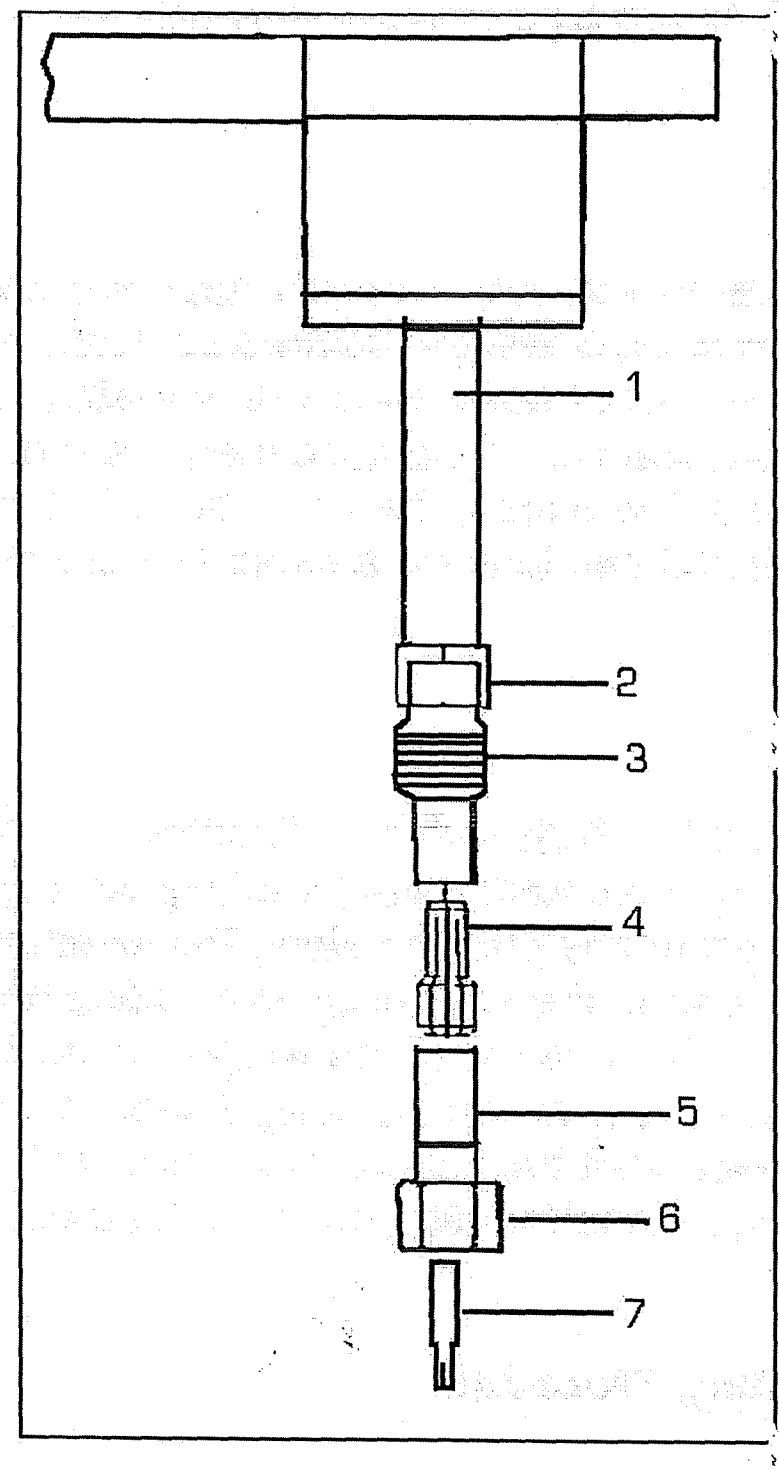


Figure 4: Illustration of the setup for the cutting tool installation. 1- Shaft; 2- Shaft bolt; 3- Threads; 4- Collet; 5- Chuck collet 5- Collet nut; 7- Cutting tool

3.2.1: Tool Type

Two different tool types are available for use with the Model 360 Abrasive Slurry Disc Cutter. The two choices are plain brass tools and diamond tipped cutting tools. Both tools are capable of cutting a wide variety of material types, but in certain instances one tool may be more useful than the other.

In general, diamond tools are used for cutting any materials where the damage at the edges does not pose any serious problems. Diamond tools are used for a wide range of metals and certain hard ceramics. Diamond tools are rather aggressive and could possibly pose some problems with microcracking and damage depending on how susceptible to cracking and chipping the specimen is.

For low damage cutting, the plain brass tools are recommended. These tools can be used with any type of abrasive media desired, from diamond to SiC. Using plain tools, most semiconductors and ceramic materials can be cut with little edge chipping, flashing, or microcracking. Cutting times are typically longer with an abrasive slurry as opposed to diamond tool cutting, depending upon the grit size used. The smaller the grit size used the longer the cutting time will be, as well as reducing the damage layer produced. Most applications are suited to using a Boron Carbide (BC) or Silicon Carbide (SiC) abrasive of around 14 microns. This is typically a good grit to use as it offers a fairly high cutting rate coupled with little damage to the specimen.

3.2.2: Tool Speed

Selecting a proper tool speed is usually dependent upon three factors; how delicate and fragile the specimen is, how fast the desired cut should be, and how much damage can be tolerated. In general, most fragile materials or materials which are very thin prior to disc cutting should be cut at slow speeds. If the specimen is sturdy and can tolerate a slight amount of chipping or damage at the edges, then high speeds are used. Typically the materials which are cut using the Model 360 can tolerate a small amount of chipping and flashing which allows for high speed cutting.

3.3: Specimen Load

Specimen load is another factor which affects the cutting process. The applied load to the tool can be adjusted by using the counter-weight adjustment knob (Figure 1, #1). By loosening this knob, l the weight can be moved to any position along the lever arm controlling the location of the tool. By increasing the applied load to the specimen, the cutting time can be reduced. The amount of reduction depends upon the location of the weight assembly.

For every 1/2" of adjustment on the top weight assembly, there is about an increase of about 70 grams. From the furthest point back (zero load), every ½" advancement of the weight is roughly 70 grams. This is a rough method for calibrating the load but is fairly accurate if the approximate load used is needed.

3.4: Standard Operating Procedure

The following method outlines the steps necessary for cutting using the Model 360. Most of the steps are referenced in previous sections and will be referred to as needed.

1. Be sure the instrument is plugged in.
2. Mount a specimen to be cut using the method described in Section 3.1.

3. If desired, align the specimen to the cutting tool using the methods described in Section 3.1.1.
4. Install the desired cutting tool to be used as described in Section 3.2.
5. Turn the instrument power switch to ON. The switch is located on the left side of the unit.
6. Adjust the cutting weight desired for the specimen being cut. This is adjusted by turning the cutting weight adjustment knob counter-clockwise (Figure 1, #1).
7. Push the START button (Figure 1, #12) to start the cutting tool rotation.
8. Adjust the speed control knob (Figure 1, #14) until the desired cutting speed is reached.
9. Adjust the time delay control knob (Figure 1, #15) until the desired limit is set. Shorter times are achieved at the minimum setting.
10. Lower the cutting tool onto the specimen by moving the latch away from the arm assembly knob (Figure 1, #2). Be sure not to let the tool slam down onto the specimen surface.
11. Apply a small amount of abrasive (or water in the case of diamond tools) to the surface of the specimen.
12. Allow the cutting tool to pass all the way through the specimen.
13. Lift the arm assembly knob once cutting has completed.
14. Remove the specimen and rinse.

M. FEI Tecnai12 TEM

In Case of an Emergency

In the case of a fire or other emergency (building evacuation),
Press the red **OFF** button on the **System Panel** (the right-hand side of the column.)
Switch the PC off.
Immediately leave the room containing the microscope and close all doors.

Logging in

Log in by typing USERNAME and PASSWORD.

The microscope user interface will start up automatically after you have logged in. If it does not, start the user interface by selecting it from the **Start** menu (or a shortcut on the Windows2K task bar).

If you need the PC of the microscope but do not require the microscope user interface (e.g. for processing data or transferring them via a network to elsewhere), you can close the user interface. This has no effect on the running of the microscope.

Starting microscope operation

1. Check the status of the panel on the right-hand side of the microscope column, continue only if the **ON** button is dark and **Vacuum** and **HT** are lit.

2. Fill the cold trap with liquid nitrogen, and check the vacuum. Proceed when **Gun/Col** Vacuum is less than 40 (30 for LaB_6).

3. Under the **Filament** control panel select desired HT from the pull-down menu, and then click on the **HT** button in the **Filament** control panel to start HT.

4. Go to **Stage** control panel and in the flip-out menu under the **Control** tab click **Holder** button.

5. Insert a specimen into a specimen holder and insert the holder into the microscope following the instructions below.

- Place and secure the specimen grid (3.05 mm diameter) in the specimen holder.
- Do not touch with bare hands the part of the specimen holder, which goes into the column (from the tip to the rubber O-ring).
- The specimen holder is introduced into the column so that the small pin on the side of the holder coincides with the slit on the Compustage where it reads **CLOSE**.
- Insert the holder half way into the column until it stops, the red light on the Compustage comes on.
- The Tecnai user interface will display a message asking for identification of the
- specimen holder. Select the type of holder from the list and press the Enter button.
- Connect the holder cable if working with the double-tilt holder.
- Wait until the specimen chamber is evacuated and the red light on the Compustage goes off.
- By holding the black handle, rotate the holder counterclockwise until stop (~ 120°) and hold it firmly. The vacuum will try to suck the holder in. Gently release the grip to allow the holder to slide all the way into the microscope column.

6. Check **Gun/Col** vacuum if less than 30 switch the filament on by clicking on the **Filament** button.

7. Wait until the filament is saturated, if the vacuum in the specimen area is good enough, click on **Col. Valves** button. The beam should now be visible on the fluorescent screen, if not reduce magnification using the magnification knob on the right-hand side pad and adjust beam intensity using the **Intensity** knob on the left-hand side pad, until light is visible on the screen.

8. Reload the most recent alignment file for the **HT** you are working at. Go to the **Alignment** control panel, from the flap-out menu select **File** and then select the alignments to be loaded.

Bright Field Imaging

1. Select magnification in the lower **M** mode (900 - 3000x) with the **MAGNIFICATION** knob.

2. In the **BEAM SETTINGS** control panel select **Spot Size** 2.

3. Center (with the left-hand track ball) and adjust (with the **Intensity** knob) beam position and intensity. The entire big fluorescent screen should be illuminated.

4. Selecting and centering of the **C2** aperture

- Make sure **SA** and the **Objective** apertures are removed from the beam (lever pointing to the right). Select desired **C2** aperture by turning the large selector knob (top aperture holder on the microscope column).
- Turn the **INTENSITY** knob to focus the beam to the smallest possible spot.

  Center the spot using the left-hand track ball.

- Turn the **INTENSITY** knob clockwise to spread the beam so that the illuminated circle is just smaller that the big fluorescent screen.
- Center the illuminated circle using the **C2** aperture mechanical shift controls (top aperture holder on the microscope column).
- Repeat the last three steps above until beam remains centered after focusing and spreading it.

5. Center (with the left-hand track ball) and adjust (with the **Intensity** knob) beam position and intensity. The entire big fluorescent screen should be illuminated. If the light on the screen is too dim go to the **Filament** control panel and increase the emission current setting using the **EMISSION** control. Do not exceed 20 μA.

6. Focus the image.

There are many ways of focusing the image. Some of the more common ones are described below.

Wobbler

- Press the **Wobbler** button on the right-hand side pad when in imaging mode
- When the objective lens is focused exactly on the specimen plane, no change in the image is apparent. However, when the objective lens is focused above or below the specimen plane there is an apparent double image
- Turn the **Focus** knob until both images overlap
- Switch the **Wobbler** off

Contrast-enhancement

In some cases (especially in biology) it is advisable to set the focus deliberately a certain amount underfocus to enhance contrast. The amount of underfocus set depends on the magnification (at high magnification an underfocus image looks blurry while the same amount of underfocus at lower magnifications may look sharp) and the degree of contrast enhancement required.

7. Center the Eucentric height following the steps below:

- **Find and center an easily recognizable feature in the specimen.**
- Activate the **α wobbler** in the **Stage** control panel, typically using 15°.
- Minimize the sideways motion of the image with the **Z** axis height control.
- **Switch the** αwobbler **off.**
- **Refocus the image as in** step 6.

8. Select and center appropriate objective aperture following one of methods below.

Method 1

- Select a suitable magnification (as required by the type of image, e.g. 5kx to 20kx for intermediate-magnification work or 100kx for high-resolution work).
- Set proper illuminating conditions (beam defocused – overfocus, i.e. clockwise with **Intensity** knob – to illuminate the whole viewing screen or just beyond the rim of the screen).
- Introduce **SA** aperture #4.
- Switch to diffraction.
- Select a camera length, using the **Magnification** knob, of approximately 500 mm.
- Focus beam to a min. spot using the **Focus** knob
- Insert the required objective aperture into the beam and center it around the central beam spot using the mechanical aperture controls.

- Switch back to imaging mode, take out the SA aperture

Method 2

- Insert the objective aperture in image mode.
- If no bright-field image is visible at all, it may be necessary to use procedure 1 for rough centering first. Otherwise, shift the aperture until there is no cut-off of the illuminated area visible (if necessary defocus/focus the beam).

For imaging at Low-magnification range select and center SA aperture

- Select a suitable magnification (around 500x).
- Set proper illuminating conditions (beam defocused – overfocus, i.e. clockwise with intensity – to illuminate the whole viewing screen or just beyond the rim of the screen).
- Switch to diffraction (LAD).
- Select a low camera length (typically the fourth of the LAD range).
- Insert the largest selected-area aperture into the beam and center it around the central beam spot using the mechanical aperture controls.

9. Find a suitable specimen area using the track-ball on the right-hand side pad.

10. Select a suitable magnification and focus the image.

11. If the image quality is not good stigmation corrections of the condenser and objective lenses need to be performed.

- Select **Help** menu,
- find the **Astigmatism** topic,
- go to **Condenser stigmation** and follow the recommended steps,
- then go to the section **Image stigmation**, select either Method 1 or Method 2 and complete the recommended steps.

Selected area diffraction

1. Obtain an image at a suitable magnification.

2. Insert the **SA** aperture and center it on the area of interest (by preference near the screen center) using the mechanical aperture controls.

If no aperture is visible upon insertion, reduce the magnification or select and center a larger aperture first before continuing on to the aperture with the required size.

3. Press **Diffraction** button on right-hand side pad.

4. Adjust the camera length to change the magnification of the SAED pattern by using the **Magnification** knob.

For selected area diffraction at Low-magnification range

1. Obtain an image at a suitable magnification.

2. Insert the objective aperture and center it on the area of interest (by preference near the screen center) using the mechanical aperture controls.

If no aperture is visible upon insertion, reduce the magnification or select and center a larger aperture first before continuing on to the aperture with the required size.

3. Press **Diffraction** button on the right-hand side pad.

4. Adjust the camera length to change the magnification of the SAED pattern by using the Magnification **knob.**

Recording an image on a negative plate

1. Obtain an image on the fluorescent screen. Make sure the details of interest are within the marks on the screen.

2. Select the workset tab containing the Plate camera **control panel.**

3. Select the appropriate parameters in the **Settings** menu of the **Plate camera** control panel.

4. Choose parameters to be printed on the plate under the **Options** menu of the **Plate camera** control panel.

5. Adjust the Intensity setting to give a normal exposure time (1 second or more).

6. Switch off ambient light and cover windows with the rubber protectors.

7. Press the **Exposure** button (the screen will be lifted, a plate loaded, the exposure recorded, the plate unloaded and the screen turned down again).

Digital Image Acquisition

US1000 HR CCD Camera

1. Launch **DigitalMicrograph**. After the application starts drag with the mouse the control window to the right-hand side monitor.
2. Turn on **GATAN CCD** camera [control box **FirstLight Digital Camera Controller**.
3. Set the **PELTIER COOLER** [control box **FirstLight Digital Camera Controller**.] switch to **COOL** position. Images can be acquired after the CCD has been cooled for at least half an hour.
4. In the menu **CAMERA** select **CCD**.
5. After the CCD has been cooled for at least half an hour collect gain reference images.
    5.1. Remove the specimen from the el. beam.
    5.2. Spread the beam so that the image of the beam is as big in diameter as the big viewing screen.
    5.3. Lift the main fluorescent screen .
    5.4. Switch **SHUTTER** control to AUTO position
    5.5. In **DigitalMicrograph** on the computer screen under pull down menu **CAMERA** with the left mouse button select **INSERT CAMERA**.
    5.6. Under pull down menu **CAMERA** select **PREPARE GAIN REFERENCE** and follow the instructions on the computer monitor [see DigMic User's Guide for more details].
6. After the gain reference has been collected insert the specimen under the el. beam. Lower the main fluorescent screen.

Obtain an image and bring the feature of interest in the middle of the main fluorescent screen using the track-ball on the right-hand side pad.

The area, which can be imaged with the CCD is slightly smaller than the area which is viewed on the small fluorescent screen.

7. Lift the main fluorescent screen.

Make sure the CCD camera is inserted.

8. To focus and adjust image.

8.1 In **DigitalMicrograph** on the computer screen in the **CAMERA VIEW** panel with the mouse select **SEARCH.**

8.2 Click with the mouse on the **TOOLS** icon in the lower-right corner of the panel, select **FULL CCD**, **BINNING – 4**, under Corrections – **Gain Normalized**.

Choose exposure time of 0.05sec. Close setup panel.

8.3. Click **START** in the **CAMERA VIEW** panel. If necessary adjust exposure time by using the **UP** or **DOWN** arrow keys on the computer keyboard. Adjust image parameters and focus.

8.4 To stop acquisition, click the stop button in the **CAMERA VIEW** panel.

9. Image recording

9.1. In the **CAMERA ACQUIRE** panel select **SETUP –> RECORD.**

9.2. Click with the mouse on the **TOOLS** icon in the lower-right corner of the panel, select **FULL CCD**, **BINNING – 1**, under **CORRECTIONS – GAIN NORMALIZED**. Choose exposure time of 1 sec. Close setup panel.

9.3. Under the **FILE** menu select **Global Info** and **Data Bar** , in the **Data Bar** panel check the box if you want scale marker automatically placed on your acquired images.

9.4. In the **CAMERA ACQUIRE** panel select **START** to acquire the image.

[see in DigitalMicrograph User's Guide for details about setting up parameters].

10. The acquired image can be saved to disk in

DM3 GATAN format [usable only with DigitalMicrograph],

in TIFF or JPG format for more details see DigMic User's Guide.

DV300 Wide-angle CCD Camera

1. Launch **DigitalMicrograph**. After the application starts drag with the mouse the control window to the right-hand side monitor.

2. Turn on **ROPER SCIENTIFIC control box.**

3. Push switch on the right-hand side of TEM viewing chamber to the left to insert the camera.

4. In the menu **CAMERA** select **DV300**.

5. Collect gain reference images.

5.1. Remove the specimen from the el. beam.

5.2. Spread the beam so that the image of the beam is as big in diameter as the big viewing screen.

5.3. Under pull down menu **CAMERA** select **PREPARE GAIN REFERENCE** and follow the instructions on the computer monitor [see DigMic User's Guide for more details].

6. After the gain reference has been collected insert the specimen under the el. beam.

Retract camera by pushing the lever switch to the right.

Obtain an image and bring the feature of interest in the middle of the main fluorescent screen using the track-ball on the right-hand side pad.

The area, which can be imaged with the DV300 CCD is as big as the area outlined by the negative markers on the fluorescent screen.

7. Insert CCD camera by pushing the lever switch to the left.

8. To focus and adjust image.

7.1 In **DigitalMicrograph** on the computer screen in the **CAMERA VIEW** panel with the mouse select **SEARCH.**

7.2 Click with the mouse on the **TOOLS** icon in the lower-right corner of the panel, select **FULL CCD**, **BINNING – 2**, under Corrections – **Gain Normalized**.

Choose exposure time of 0.05sec. Close setup panel.

7.3. Click **START** in the **CAMERA VIEW** panel. If necessary adjust exposure time by using the **UP** or **DOWN** arrow keys on the computer keyboard. Adjust image parameters and focus.

7.4 To stop acquisition, click the stop button in the **CAMERA VIEW** panel.

9. Image recording

9.1. In the **CAMERA ACQUIRE** panel select **SETUP –> RECORD.**

9.2. Click with the mouse on the **TOOLS** icon in the lower-right corner of the panel, select **FULL CCD**, **BINNING – 1**, under **CORRECTIONS – GAIN NORMALIZED**. Choose exposure time of 1 sec. Close setup panel.

9.3. Under the **FILE** menu select **Global Info** and **Data Bar** , in the **Data Bar** panel check the box if you want scale marker automatically placed on your acquired images.

9.4. In the **CAMERA ACQUIRE** panel select **START** to acquire the image.

[see in DigitalMicrograph User's Guide for details about setting up parameters].

10. The acquired image can be saved to disk in

DM3 GATAN format [usable only with DigitalMicrograph],

in TIFF or JPG format for more details see DigMic User's Guide.

Removing a specimen holder from the microscope

- Close the column valves.
- Go to **Stage** control panel and in the flip-out menu under the **Control** tab click **Reset Holder** button. The red **CompuStage** light should go off.

- **Desaturate** the filament 5 steps if you are changing sample **(LaB_6). Turn off** the filament if you are ending the session
- Pull the holder as far out of the CompuStage as it will go, then rotate it clockwise as far as it will go (about 120 degrees).
- Carefully extract the holder from the airlock (you have to pull against the vacuum remaining in the airlock).
- For double-tilt holders disconnect the cable plug.
- If you are changing samples be sure that the once the specimen holder is inserted into the beam, the **column pressure is below 30** before **resaturating (LaB_6)** the filament.

Ending a microscope session

DO NOT select **SHUT DOWN** the **COMPUTER** because that will shut down the microscope completely!

1. Close the gun valves by clicking on the **Col .Valves** in the **Gun** Control panel.
2. Switch the filament off by clicking on the **Filament** button.
3. Turn off the high tension. By clicking the **High Tension** button.
3. In the Windows2K **Start** menu, select **Log Off.**
    - When you log off from the microscope (close the Tecnai User Interface) you will be asked if you wish to save system settings or not.
    - Select **Yes** if you wish to save the column parameters like spot sizes, intensities, knob settings defined by you, plate camera, and vacuum settings.
    - The 'system settings' do not include the changes you have made to the Tecnai User Interface layout like the selection of control panels and their arrangement over the tabs and the color selections made in Workspace Layout. Those changes are saved immediately as you make them.
4. Record your session in the log book.

Electronic sign-up

1. You will receive an email with your user name and password.
2. Using a web browser go to http://faces.ccrc.uga.edu/
3. Login by typing:
   CFAMM in the GROUP field,
   in the USER NAME field type in your logon name,
   in the PASSWORD field type in your password
   Click the GO button.
4. Under "select a resource", choose the instrument you want to sign-up for, in your case "TEM T12"
5. By clicking the month and dates, choose the date you wish.
6. Click the button to the right of the time you wish to start, then click the button to the right of the time you wish to end.
7. Click the "reserve this time" button when you have chosen the date and times you desire.

8. Confirm this in the box that comes up.
Do not leave messages for in the space available, email bozhilov@ucr.edu directly if there is an issue that needs attention.
9. Logoff - you are scheduled. You can reschedule if you need to - follow the above directions, at #6 click on the button for the time you have already scheduled (the date must already be set appropriately), you will be given the option to cancel the appointment.

NOTE- IF YOU CANCEL AN APPOINTMENT LESS THAN ONE WORKING DAY IN ADVANCE, YOU WILL BE CHARGED FOR THE TIME OF YOUR RESERVED HOURS.

N. SORVALL GLASS KNIFE MAKER - INSTRUCTIONS

I - Wash glass rectangles with alconox and scrub brush.

II - Reducing glass rectangles to 2.54cm (1 in) squares.

1 - Remove plastic cover.

2 - Center glass using a ruler on the top plate against the back stop plate. Support ends of glass rectangle if the rectangle is very long otherwise excessive pressure will be placed on the score line.

3 - Check glass to ensure that the manufacturer's score is down (This side is identified by minute ridges along the edge)

4 - Turn the handwheel clockwise while holding the glass in place. After the bumpers have made contact with the glass, complete on more full turn of the handwheel.

The glass should be held securely. Try to rock the anvil to see if the glass is secure. If the anvil can be moved, continue turning the hand wheel RAPIDLY until the anvil is tight.

5 - Pull the plunger adjustment shaft out to prepare for the short stroke.

6 - Pull the scoring shaft all the way out. Depress the button and push the scoring shaft back with one even stroke to score the glass.

7 - SLOWLY, GENTLY, and CAREFULLY turn the breaking control knob clockwise until the glass breaks.

8 - Turn the handwheel counterclockwise to raise the clamp and anvil

NEVER TURN THE BREAK CONTROL KNOB COUNTERCLOCKWISE IT WILL RESET AUTOMATICALLY
NEVER RAISE THE CLAMP ARM TO ITS HIGHEST POSITION

9 - Remove glass by placing your fingers on top of the glass and sliding the pieces toward you.

Repeat all steps until glass is broken into 2.54 cm squares.

III - Preparation of Knives

1 - Inspect the corners and sides of the square carefully. Select the corner that is the smoothest (with the fewest score marks) and the most nearly perpendicular to the 2,54cm square.

2 - Place the glass square on the top plate with the selected corner seated in the back stop plate registration notch. The scored edges of the glass square should now be up

3 - Push the positioner slide lightly against the corner of the glass facing the operator.

4 - Hold the positioner slide lightly in place while turning the handwheel clockwise to clamp the glass to the top plate.

5 - Push the plunger adjustment shaft IN for a long stroke.

6 - Pull the positioner back so it is flush against the top plate surface.

7 - Pull the scoring shaft all the way out. Depress the button and push the scoring shaft back in one even stroke.

8 - Place plastic cover over the instrument.

9 - SLOWLY AND CAREFULLY turn the breaking control knob CLOCKWISE until the glass breaks.

10 - Remove the plastic cover.

11 - Turn the handwheel counterclockwise to raise the clamp arm and anvil.

NEVER TURN THE BREAK CONTROL KNOB COUNTERCLOCKWISEIT WILL RESET AUTOMATICALLY
NEVER RAISE THE CLAMP ARM TO ITS HIGHEST POSITION

Inspect each glass knife.

The outer one-third of the knife edge that parallels the stress line will be the sharpest section.

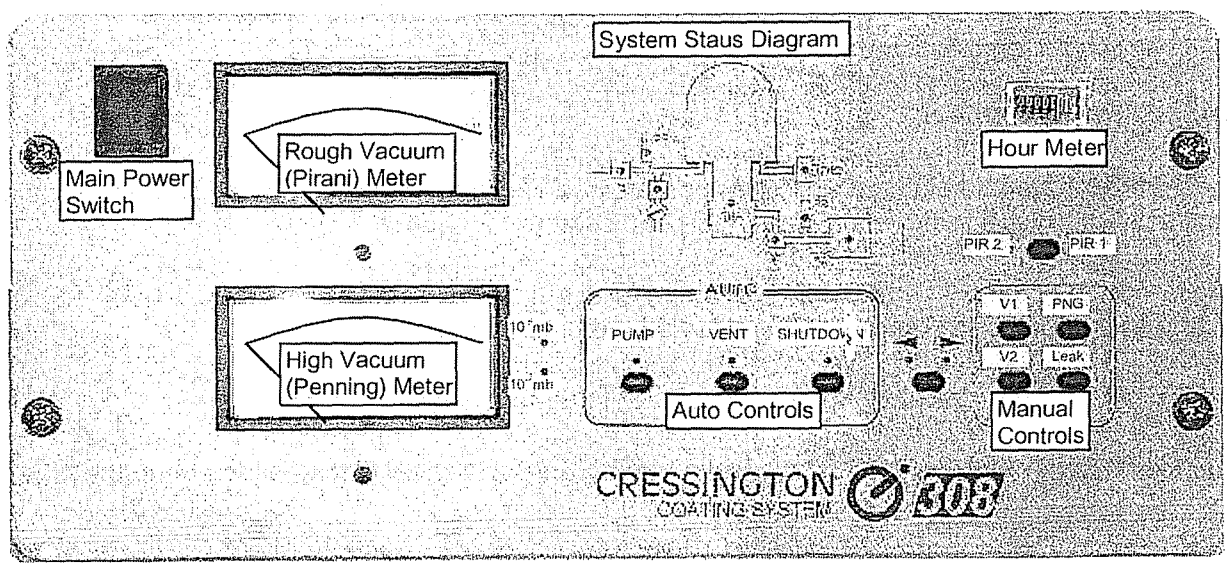
The final test of the acceptability of the glass knife is to examine the knife edge for "whiskers". This is done under a binocular stereo microscope with bright field illumination from above against a dark background.

O. Cressington 308R Vacuum Evaporator

Basic Operation

3.1 Front Panel

The Cressington 308 pump station front panel is illustrated below.



Cressington 308, Pump Station Front Panel

The main power switch pressure meters, system status diagram, hour meter, and auto and manual controls are indicated, and their functions are as follows:

3.1.1 Main Power Switch

On operation of the power switch the Cressington 308 pump station will automatically enter auto pump mode. From this mode it is not possible to use the shutdown function until the turbo pump has attained full rotational speed. The LED marked "TMP" flashing indicates turbo pump acceleration on the system status diagram. Once the turbo is at speed the LED is permanently on.

It is possible to vent the system or select manual operation to access further functions during turbo pump acceleration.

It is recommended that the Cressington 308 pump station be either shutdown or vented prior to switching off. However, switching the power off under high vacuum mode, whilst not recommended will not cause damage.

3.1.2 Pressure Meters

The rough vacuum meter indicates the pressure in either (a) the chamber being monitored by Pirani gauge head 2 or (b) the backing line monitored by Pirani gauge head 1. The push button marked "PIR 2" to the left and "PIR 1" to the right may be used to manually toggle between heads whether in Auto or Manual mode. The corresponding LED on the system status diagram is illuminated to indicate which

pressure is being displayed. These LEDS will flash until the factory set-points have been reached.

The high vacuum meter indicates the pressure in the main chamber as measured by the Penning gauge. The Penning gauge is enabled when PIR 2 achieves a factory- defined set point. Once enabled the "10^{-5} mb" range indicator LED is illuminated until a pressure of $7x1^{-6}$ mbar ($5x10^{-3}$ mTorr) is reached. In this range the pressure can be read from the top scale of the meter. Below $7x1^{-6}$ mbar ($5x1^{-3}$ mTorr) and until the pressure rises above $9x10^{-6}$ mbar ($6x10^{-3}$ mTorr) the "10^{-7} mb" range indicator LED is illuminated and the pressure can be read from the bottom scale of the meter.

3.1.3 System Status Diagram

The status of the system during all functions is indicated on a block diagram on the front panel of the Cressington 308. Illuminated LEDS indicate that a system component is active, with flashing LEDS indicating that some set point or speed has not yet been achieved. All valves are normally closed, so an illuminated valve LED indicates that the valve is open.

3.1.4 Hour Meter

An hour meter is provided to indicate the total number of hours of operation of the system. This is primarily for the provision of service of the vacuum pumps according to their manufacturers' instructions.

3.2 Auto Control Functions

3.2.1 PUMP

When switched on the Cressington 308 will default t0 auto pump mode. This will be indicated by the pump LED being illuminated. The initial conditions are as follows:

V1, V2, Leak Isolation Valve closed;
RP, TMP, PNG off;
PIR 1 selected (LED flashing).

The system software will perform a self-check process, which may take up to a minute before the following pump down sequence begins:

TMP started (no backing - LED flashing);
RP started with V1 shut. Backing line evacuated to PIR 1 set point;
PIR 2 selected (LED flashing), V1 opened and chamber evacuation begins;
PIR 2 set point reached (LED stops flashing) and PNG activated;
TMP reaches nominal speed (LED stops flashing).

NOTE: It is acceptable to change Pirani selection during this process without affecting the automatic control. Pirani selection denotes the pressure indicated only, whilst the software will monitor the pressure required by the pump–down process. The system status diagram will indicate the pressure being displayed if different from that being used by the software, but if not changed the pressure displayed will default to the software selection.

3.2.2 VENT

The Vent command allows the Cressington 308 pump station to be vented to atmospheric pressure. The turbo–pump exhaust isolation valve is closed and the turbo-pump turned off before the vent valve is opened to allow a gradual vent. Venting times of the order of 3 minutes can be expected, though this is

strongly dependent on vent gas pressure.

On pressing "VENT" an audible 6 second warning (consisting of 3 one second beeps followed by 6 half second beeps) is given before venting commences. During this time pressing any Auto command push—button will cancel the vent command. After this period the vent push-button can be used to open and close the vent valve without reinitiating the pump function. Thus, once the system has been fully vented and is open to atmosphere, the vent push—button can be used to close the vent valve to conserve the vent gas supply.

The vent command does not affect the status of the rotary pump. Thus, if vented from pump the rotary pump will be isolated and continue to evacuate the backing line. If vented from shutdown, however, the rotary pump will remain off.

3.2.3 SHUTDOWN

The shutdown command allows the Cressington 308 pump station to be stored under a partial pressure (approx. 1 mbar/ 1 Torr) of vent gas. This minimizes operating hours and thus extends life between servicing, whilst providing for very rapid recovery to high vacuum. Indeed storage under a low pressure of dry vent gas can improve vacuum performance.

The shutdown function isolates the turbo—pump backing line by closing V1 and shuts down the rotary pump, The turbo-pump is powered down and the vent valve pulsed open to allow a small volume of vent gas into the chamber and turbo—pump.

On pressing "SHUTDOWN" an audible 6 second warning (consisting of 3 one second beeps followed by 6 half second beeps) is given before shutdown commences. during this time pressing any Auto command push-button will cancel the shutdown command. The pump station may be turned off at the main power switch within a second or so after this period.

Manual Control Functions

The push-button situated between the Auto and manual controls can be used to toggle between auto and manual control. The two LEDS situated above the push-button indicate which control is selected. The following functions may be operated manually:

V1 Opens and closes the turbo-pump exhaust isolation valve.

PNG activates and do—actives the Penning gauge. The Penning gauge should be do-activated when the leak valve is open.

V2 Opens and closes the vent valve.

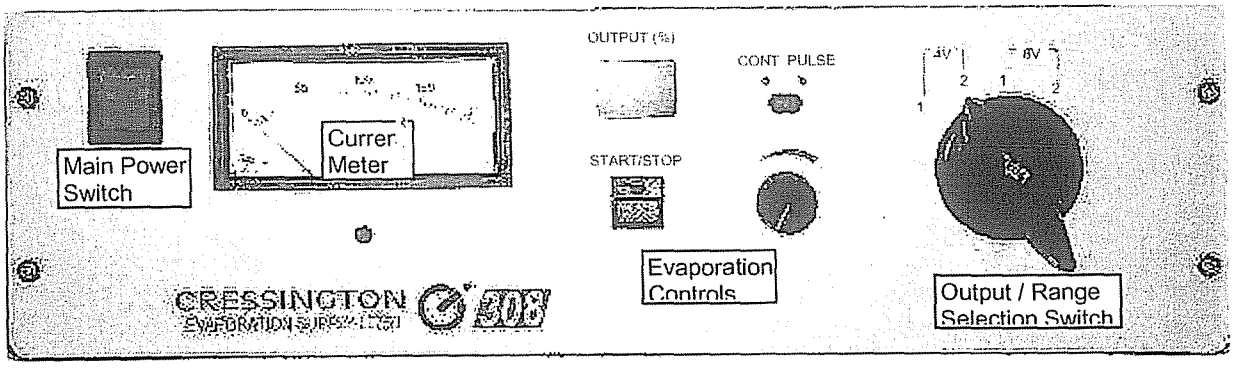
Leak Opens and closes the leak shutoff valve. The leak rate is set by the needle valve on the back panel of the base unit.

LT 750 Evaporation Supply

Operation

3.1 Basic Operation

The LT 750 front panel is illustrated below:



Cressington 308, LT 750 Front Panel

The main power switch, output current meter, evaporation controls and output / voltage range switch are indicated, and their functions are as follows:

3.1.1 Main Power Switch

The main power switch activates the power supply. However, no operation is possible until the vacuum switches in the current feedthroughs are closed. This is a safety feature only. The vacuum switches typically close at around 100mbar (100Torr) at which pressure evaporation should not be performed. Typically an evaporation pressure of better than 10^{-5}mbar (0.01mTorr) is recommended, depending on process.

3.1.2 Output Current Meter

The output current meter indicates the current being supplied to the Head selected. The meter has a full-scale deflection of 200A but typically a trip current of around 185A limits the output. The scale is calibrated in Amps so can only reach half-scale deflection when the 8V range is selected.

3.1.3 Evaporation Controls

The LT 750 may be operated in either continuous or pulse mode, and these are selected by toggling the "CONT. PULSE" push-button and the selected mode is indicated by the LEDs above and to either side of this.

When CONTINUOUS is selected the "START/STOP" switch turns on / off the current output. When PULSE is selected the output is only on whilst the "START/STOP" switch is depressed. The "START/STOP" switch incorporates an "enabled" LED, which is on when the vacuum switched in both Head 1 and Head 2 are closed, The output cannot be turned on whilst the coating system is at atmospheric pressure and the LED is off.

The evaporation supply voltage is set using the VOLTS knob and this can be used to adjust the evaporation current in both modes. The LED display marked "OUTPUT (%)" indicates the applied voltage as a percentage of the selected voltage range. For example 4.5V would be shown as 56% with an 8V output range selected.

3.1.4 Output! Range Selection Switch

The LT 750 has two outputs, either of which may be used with either a 4V or 8V full-scale output voltage range. These are selected by switching between taps on the high current transformer so it is the 4V ranges that offer a maximum current limited to 185 Amps, whilst the BV ranges are limited to around 90-95 Amps. The range and output Head are selected using the Output / Range selection switch at the right of the front panel. Head 1 is the output to the left at the rear (viewed from above), and Head 2 the right.

The 4V range should be selected for low resistance sources such as thermal evaporation boats, whilst higher resistance techniques such as filament evaporation or Bradley carbon evaporation will require an 8V range.

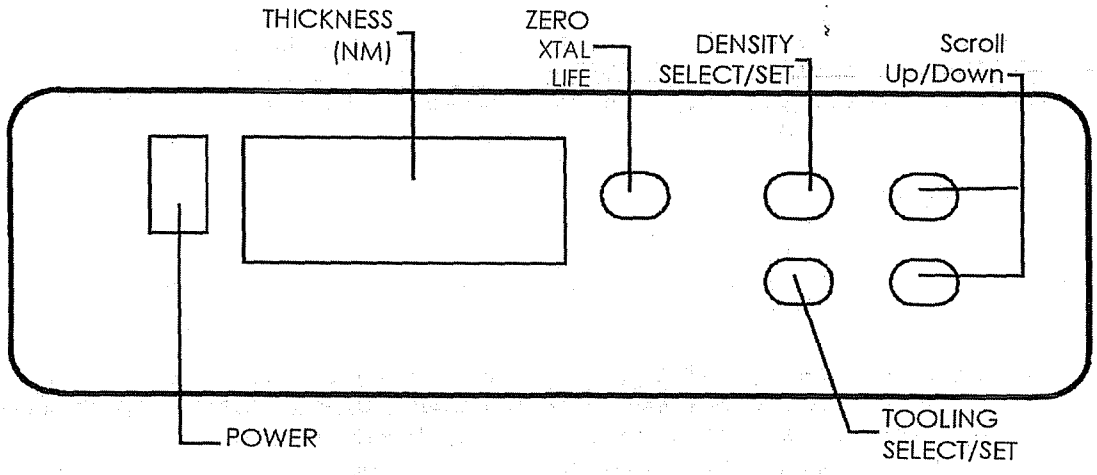
MTM-10 Thickness Monitor

General

The MTM—10 thickness monitor works on the principle of the quartz crystal microbalance When material is deposited on the oscillating quartz crystal its frequency is decreased. The frequency change is used to calculate the Elm thickness using the density (gm/cm^3) of the deposited material. The thickness monitor calculates the thickness of deposit every tenth of a second and displays the result in nanometers on the digital THICKNESS display.

Description

The thickness monitor calculates the thickness of deposit every tenth of a second and displays the result in nanometers on the digital THICKNESS display. It detects the frequency shift and uses the DENSITY and TOOLING values keyed into memory. Four sets of values can be stored under DENSITY and TOOL/NG4 The front panel of the control unit is shown below.



To display the currently selected value of material density or tooling factor, press the relevant key and the value will appear on the THICKNESS display. To select another of the four values scroll up or down to the other values using the scroll keys. To adjust that value, hold down the relevant key for two seconds until the period flashes and scroll up or down using the scroll keys.

The measuring crystal can only accept a finite amount of sputtered material before becoming overloaded. The amount of offset from its original frequency (6.0lVlHz) is displayed (in KHz) when XTAL LIFE is pressed for two seconds. The expected life of a crystal will depend on (i) amount of deposited material and (ii) stress level of deposited material. When the crystal ceases to oscillate the display shows "FAIL". To reset the thickness between runs press ZERO.

The interpretation of the thickness monitor reading during evaporation is not straightforward. The monitor is on a sensitive scale and the effects of radiation heating become obvious. The effect of heating is to increase the frequency of oscillation (it responds very rapidly to radiation) and a negative reading is obtained; the effect of depositing material is to decrease the frequency and give a positive reading. In the early stages of deposition the net reading will be negative and in the late stages it will be positive but under-reading.

Installation

Carefully unpack the MTM-10 control box and associated parts. Take care not to damage the delicate crystal in the crystal head. A complete thickness monitor outfit comprises:

(1) a crystal head
(2) crystal head cable
(3) a vacuum feedthrough
(4) an oscillator unit near the vacuum feedthrough
(5) BNC type cable
(6) power cord
(7) the MTM-10 control box.

IMPORTANT
CHECK THE SERIAL NUMBER LABEL ON THE BACK PANEL TO ENSURE THAT THE SUPPLY VOLTAGE IS SUITABLE

Fit a suitable 3-pin plug to the power cord for the MTM-10 control box.

IMPORTANT

THE POWER CORD FOLLOWS THE EUROPEAN CONVENTION:
LIVE = BROWN
NEUTRAL = BLUE
GROUND = GREEN I YELLOW

AND THE PLUG SHOULD BE WIRED ACCORDINGLY.

Mount the crystal head and feedthrough on a suitable position in the deposition system. Mount the oscillator unit outside the vacuum chamber on the BNC bulkhead of the vacuum feedthrough. The distance between crystal head and oscillator unit should be as short as possible and should not exceed 40cm. Use the BNC cable to connect the oscillator unit to the MTM-10 thickness monitor

Quartz Crystal Changing

When the thickness monitor crystal is either over-loaded or loaded with stressed material it will cease to oscillate and the display will read "FAIL". At this stage it will need to be changed.

IMPORTANT
TAKE SAFETY PRECAUTIONS WHEN WORKING WITH POTENTIALLY HAZARDOUS MATERIALS.

(a) Switch off the thickness monitor system.
(b) Secure system before venting the vacuum chamber
(c) Loosen the 2 small screws in the side of the head. This will allow the crystal holding cap of the head to be separated from the base.

(d) Carefully remove the cap leaving the crystal balanced on the spring. Note the position. Now replace the used crystal with a new crystal. Carefully replace the cap.
(e) Push gently to compress the spring. Retighten the small screws.
(f) Repump the system

Trouble shooting

MTM-10 display shows "FAIL"

Solution: Change crystal
Check cables connections
Check contact between crystal and crystal head

MTM-10 display shows "or'

Reason: Static discharge damaged IC's inside the control boxUnit has to be sent back for repair and re-calibration

3 — Make sure ambient temperature is not below 15°C (60 F)

Consumables and spare parts

93008 Thickness Monitor Crystals, pkg/3
93009 Thickness Monitor Crystals, pkg/10

Specifications

V For MTM-10:

MTM-10 Microprocessor based
4 digit display
Push button zero
6Mhz crystal with lifetime check

Memory	Four settings storing Density and Tooling factor
Update rate	10 Hz
Thickness range	0.0 - 999.9nm
Resolution	better than 0. 1 nm
Density range	0.50 — 30.00 gm/cm^3
Tooling factor range	0.25 - 8.00
Error Messages	Fail I or

Tooling Factor

The formula for Tooling Factor is:

$$TF = \left\{ \frac{d\ Monitor}{d\ Specimen} \right\}^2 \left\{ \frac{\sin\varnothing_s}{\sin\varnothing_m} \right\}$$

Where: d Monitor is the distance between the source and the crystal monitor.
d Specimen is the distance between the source and the specimen.
$\varnothing_s$ is the angle between the specimen and the source.
$\varnothing_m$ is the angle between the monitor and the source.

P. FEI XL30-FEG SEM

The XL30 user interface consists of two monitors. The left-hand side monitor displays the desktop of the EDAX PC, where user files should be stored. The right-hand side monitor displays the desktop of the XL30 PC, on which the microscope image is displayed. The two computers can be accessed using a single mouse and keyboard.

Do not connect any devices to the XL30 PC!

1. Starting a user session

1. Check the status of the SEM control panel on the left side (under the **Polaroid** camera) and proceed only if: **ON** button is dark, **STANDBY, OFF, VACUUM** and **HIGH TENSION** buttons are lit. In case button configuration is different, stop and contact staff personnel immediately.
2. If desired plug in your USB device to the front of the **EDAX PC**.
3. Make sure the red LED in **position 2** on the **SERVEVIEW** switch is highlighted, if not press one of the red buttons on the switch to set it in **position 2**.
4. Login to the **XL30 PC** on the right-hand side monitor using your own username and password.
5. After login, at the password prompt on the right-hand side monitor type: **user**.
6. If **XL30 Control** does not start automatically, find the icon **XL30 CONTROL** on the desktop and launch the program by double-clicking the icon.

2. **Inserting a specimen**

1. in control area **SETTINGS**, under submenu **BEAM** make sure **kV** button is off
2. in control area **SETTINGS**, under submenu **VACUUM** press **VENT** button and confirm
3. open Nitrogen flow by turning fully counterclockwise the small black plastic knob on the pressure reducing system on the black metal cylinder with compressed Nitrogen gas
4. wait until chamber reaches atmospheric pressure, then pull on the gray handle to open the chamber.
5. stop Nitrogen flow by closing the small black plastic knob

6. using lint free gloves and/or tweezers, place a specimen into the stage holder and tighten the set screw.

7. VERY IMPORTANT! Check the clearance between the upper surface of the specimen and the final lens polepiece, respectively the solid state detector, and if necessary adjust the height and/or position of the specimen using the manual stage control knobs

8. close the chamber, press the door towards the column with hand, under submenu **VACUUM** click **PUMP** while pressing on the door, hold for about 5 sec. - the automatic pumping sequence starts.

3. **Obtaining an image**

1. under menu bar **MAGNIFICATION** select lowest magnification

2. under menu bar **BEAM** choose desirable kV and **SPOT SIZE 3**

3. select desirable final lens aperture by using the mechanical controls on the microscope column (above the SE detector).

The following apertures can be selected:

*position 1	Au 30 μm;
position 2	Pt 30 μm;
position 3	Pt 50 μm; and
position 4	Pt 100 μm.

**** Position 1 Au 30 μm aperture should be used only at accelerating voltages ≤ 5 kV.***

4. under menu bar **SCAN** select **TV**

5. under menu bar **DETECTORS** select **SE**

6. under menu bar **DETECTORS** select **SETUP** and set bias voltage to +300V

7. under menu bar **FILTER** select **AVERAGE 4**

8. in control area **SETTINGS** submenu **VACUUM** check pressure in chamber (top reading). If the pressure is less than **$8x10^{-5}$ mBar** go to control area **SETTINGS** submenu **BEAM,** click **kV** button to turn on the electron beam.

9. under submenu **VIDEO** adjust contrast and brightness by sliding the corresponding buttons with the cursor

10. bring the area of interest in the middle of the screen using the manual stage controls

11. adjust magnification if necessary and focus specimen by pressing and holding the right mouse button and sliding the cursor in horizontal direction.

4. Setting the Working Distance

1. select appropriate magnification.

2. focus image precisely and read the distance displayed in millimeters in the data bar under **WD.**

3. using the right mouse button change focus to the desired **WD** value, image could go out of focus

4. turn the **Z** control knob on the specimen stage (clockwise to decrease the working distance, counterclockwise to increase it) until image appears in focus.

5. Optical alignments

Necessary to conduct after spot size, kV, working distance, objective aperture, or specimen have been changed.

5.1 **Objective aperture centering**

1. select magnification 200
2. if necessary adjust contrast and brightness and focus
3. press **CONTROL-X** on the keyboard - small red cross appears in the center of the screen
4. select an easy recognizable feature on the sample, bring it under the red cross by using the stage controls, and focus
5. under control area **SETTINGS,** submenu **BEAM** check the box **LENS MODULATOR**
6. press **CONTROL-X** on the keyboard - small red cross disappears
7. minimize the movement of the selected feature by turning the objective aperture displacement controls (knobs on the side of the column, above the SE detector), pulsation of image should remain even with aligned aperture.
8. deselect the box **LENS MODULATOR** under control area **SETTINGS**, submenu **BEAM**.
9. select magnification 5000
10. repeat steps 2 to 8 and continue with astigmatism correction

5.2. **Astigmatism correction**

1. select area containing isometric objects or features with submicron size
2. select magnification 40 000
3. adjust contrast and brightness under control area **SETTINGS,** submenu **VIDEO**
4. focus image
5. go to control area **IMAGING,** with the left mouse button click on the **STIGMATOR** box with cross and hold, a cross appears on the screen, by sliding the mouse correct the image astigmatism
6. check focus and repeat correction until satisfactory.

6. Optimizing the image

1. under menu **SCAN** select **TV,** and choose low magnification (~200x)
2. bring close to the middle of the screen and orient the area of interest by using the **X**, **Y** and **R** knobs located on the side of the specimen chamber
3. select desired magnification and choose detail of interest
4. increase magnification by factor of two, focus, and decrease magnification.
5. under menu bar **FILTER** select **STANDARD DEFINITION** or **HIGH DEFINITION** according to your preference.
6. under menu **SCAN** select **SCAN PRESETS** and set up desired scanning speeds and frame sizes. Choose **484** lines per frame for **STANDARD DEFINITION** and **968** lines per frame for **HIGH DEFINITION.** Set up **SLOWSCAN1**, **SLOWSCAN2**, **SLOWSCAN3** and **PHOTOSCAN**, click **OK** after completion.
7. under menu bar **SCAN** select **FULL FRAME** and an appropriate slowscan.
8. click the **VIDEOSCOPE** icon

9. adjust the contrast and brightness of the image using the brightness and contrast controls in submenu **VIDEO**, keep the signal amplitude within the shown broken-line boundaries

10. click the **VIDEOSCOPE** icon to deselect videoscope.

7. Image output

1. After completing procedure *6. Optimizing the image*, click **FREEZE IMAGE** icon

2. Under menu bar **IN/OUT** select **DATABAR SETUP**, enter or delete the annotation, which will appear on your image. Click **OK.**

3. Saving digital image files. Filename and directory names should be a single word and no longer than 8 characters.

3.1. Storing in the USERS directory on the EDAX PC hard disk:

Under menu bar **IN/OUT** select **IMAGE**. Check the box **DATABAR** if you want to store the databar annotation. To store the image enter path, filename, image format, e.g. **F:\NAME.TIF** and click **SAVE.**

3.2. Storing to a USB device:

Under menu bar **IN/OUT** select **IMAGE**. Check the box **DATABAR** if you want to store the databar annotation. Enter path, filename, image format, e.g. **E:\NAME.TIF**, and click **SAVE.**

4. After saving the files in .TIF format you must resize them using **XL-STRETCH.**

If saving to the hard disk one should create his/hers own directory on drive F:(Directory USERS **on the EDAX PC.** Do not store images on drive C of the XL30 PC!!!

8. Removing the specimen

1. In control area **SETTINGS**, under submenu **BEAM** turn **KV** off
2. In control area **SETTINGS**, submenu **VACUUM** press **VENT** button and confirm
3. open Nitrogen flow by turning fully counterclockwise the black plastic knob on the pressure reducing system on the black metal cylinder with compressed Nitrogen gas
4. wait until chamber reaches atmospheric pressure and then open the chamber
5. stop Nitrogen flow by closing the black plastic knob
6. release the tightening nut, using lint free gloves and/or tweezers take out the specimen from the stage holder
7. close the chamber, press the door towards the column with hand and click **PUMP** while pressing the door, hold for about 5 sek. - automatic pumping sequence starts.

9. Concluding the session

1. Fill out the log book, recording the time of the session, user name/advisor name, emission current, accelerating voltage.
2. Under menu bar **MAGNIFICATION** select lowest magnification
3. Under menu bar **BEAM** choose **SPOT SIZE 3**
4. Under menu bar **SCAN** select **TV**
5. Under menu bar **FILTER** select **AVERAGE 4** or **LIVE**

6. Select final lens aperture position 3 using the mechanical controls on the column.

7. Close the **Microscope Control** application.

8. Logoff from your user account on the **XL30** PC (right-hand side monitor).

Do not shutdown the XL30 PC.

10. Acquiring and saving digital images in SCANDIUM in Extra High Definition

1. Start **SCANDIUM** software

2. Go back to **XL30 Control** software, under menu bar **FILTER** select **HIGH DEFINITION**.

3. After selecting and optimizing your image go to menu bar **SCAN** -> **SCAN PRESETS**. Select **XHD** and choose linetime and frame size.

4. Under menu bar **IN/OUT** select **IMAGE,** enter path and filename, select **XHD** and then click **SAVE&SCAN.**

5. After the image is saved go to the **SCANDIUM** window, in the browse menu on the left click image icon **1**.

6. Select **IMAGE** from the menu bar and click **SNAPSHOT**, the image will be acquired.

7. Go to the **DATABAR** button and turn it **ON** or **OFF** according to your preferences. In the lower right corner of the screen click the **SETTINGS** icon and choose the information you want to be displayed in the databar.

8. To save the image, go to **FILE** -> **SAVE AS**, select desired file format and name. Save image to dirve **F:** or on your USB device (drive **E:**).

Do not store images on drive C. *The images are in proper aspect ratio it is **NOT** necessary to re-size them.*

11. Polaroid photograph

1. Do procedure *6. Optimizing the image*

2. Under menu bar **IN/OUT** select **CHANGE**, see that correct film parameters are elected, change if necessary, when finished press **EXIT**

3. Under menu bar **IN/OUT** select **DATABAR**, enter or delete whatever is necessary. Press **OK**

4. Load film in Polaroid camera, move control arm to L. Hold envelope near center with POLAROID facing you; insert halfway into holder, shift grip to end of envelope and push in all the way; withdraw envelope (not completely).

- 5. Under menu bar **IN/OUT** select **PHOTO!**
- 6. After exposure is done move control arm on Polaroid camera to P; pull envelope completely out of holder at medium speed, without stopping or hesitating; process for time given in film instructions, then open envelope and separate print from negative.

Electronic sign-up

1. After you receive an email with a password, go to http://faces.ccrc.uga.edu/
3. Login by typing:
   CFAMM in the GROUP field,

in the USER NAME field type in your logon name,
in the PASSWORD field type in your password
Click the GO button.

4. Under "select a resource", choose the instrument you want to sign-up for, in your case SEM-XL30
5. By clicking the month and dates, choose the date you wish.
6. Click the button to the right of the time you wish to start, then click the button to the right of the time you wish to end.
7. Click the "reserve this time" button when you have chosen the date and times you desire.
8. Confirm this in the box that comes up.
   Do not leave messages in the space available, email bozhilov@ucr.edu directly if there is an issue that needs attention.
9. Logoff - you are scheduled. You can reschedule if you need to - follow the above directions, at #6 click on the button for the time you have already scheduled (the date must already be set appropriately), you will be given the option to cancel the appointment.

NOTES - IF YOU CANCEL AN APPOINTMENT LESS THAN ONE WORKING DAY IN ADVANCE, YOU WILL BE CHARGED FOR 2 HOURS OF USE.
MINIMUN CHARGE FOR SEM TIME IS 1 HOUR, INCREASING BY INCREMENTS OF ½ HOUR.

Note: Digital images need to be resized to view with non-Philips software.

Standard definition:	712x484 resize to:		712x532 pixels
High definition:	1424x968	->	1424x1064 pixels

You can use **XL-STRETCH** to resize digital image files.

VII. Working Alone

Working with hazardous chemicals alone, especially after regular hours in a laboratory poses an additional risk to life and property. If an unanticipated event occurs and the researcher is incapacitated, available safety equipment, such as emergency showers, fire extinguishers, or antidotes, become worthless to provide emergency assistance to injured individuals or to mitigate the losses incurred. Each Laboratory needs to incorporate into its Individual Chemical Hygiene Plan a working alone procedure suited to the needs of the lab.
Suggestions for provisions that may be incorporated are:

1. Prohibiting working alone in laboratories, or prohibiting working alone for certain procedures.
2. Buddy System
   a. Arrange with someone working nearby to periodically check on one another.
   b. Arrange to take scheduled breaks with another researcher working nearby.
   c. Arrange to periodically check in with another person telephonically, or through radios.
3. Remote monitoring by Web Cam or Closed Circuit Television.

VIII. Unattended Operations

A. Unattended electrical heating equipment should be provided with a manual reset overtemperature shutoff switch, in addition to normal temperature controls.
B. Unattended operations that depend on a flowing cooling water supply should be provided with a manual reset shutoff device that will shut off the heat supply upon loss of cooling water.

C. Arrangements should be made, where possible, to periodically check unattended operations for abnormal conditions.
D. For operations left running after normal hours, leave the lights on and leave a sign on the door indicating that an unattended procedure is in progress. The sign should also give emergency phone numbers in case of incident.

IX. Glassware

(from Prudent Practices for Handling Hazardous Chemicals in Laboratories. National Research Council. Washington, D.C.: National Academy Press, 1981. pp. 25-26.)

Accidents involving glassware are a leading cause of laboratory injuries.

1. Careful handling and storage procedures should be used to avoid damaging glassware. Damaged items should be discarded or repaired.
2. Adequate hand protection should be used when inserting glass tubing into rubber stoppers or corks, when inserting glass pipets into dispensing bulbs, or when placing rubber tubing on glass hose connections. Tubing should be fire polished or rounded and lubricated, and hands should be held close together to limit movement of glass should fracture occur. The use of plastic or metal connectors should be considered.
3. Glass-blowing operations should not be attempted unless proper annealing facilities are available.
4. Vacuum-jacketed glass apparatus should be handled with extreme care to prevent implosions. Equipment such as Dewar flasks should be taped or shielded. Only glassware designed for vacuum work should be used for that purpose.
5. Hand protection should be used when picking up broken glass. (Small pieces should be swept up with a brush into a dustpan.)
6. Proper instruction should be provided in the use of glass equipment designed for specialized tasks, which can represent unusual risks for the first-time user. (For example, separatory funnels containing volatile solvents can develop considerable pressure during use.

X. Laboratory (Fume) Hood Usage

The Environmental Health and Safety Office will inspect laboratory hoods annually to determine proper function and adequate face velocity. The following are considerations for the laboratory hood users that will facilitate optimum hood performance.

A. Environmental Health and Safety should be consulted for new hood installations.
B. Laboratory hoods should not be relied upon to provide explosion (blast) protection unless specifically designed to do so.
C. When perchloric acid is evaporated or heated above ambient temperatures in a laboratory hood, the hood should be specifically designed for perchloric acid.
D. For new installations or modifications of existing installations, fixed electrical services and their controls should be located external to the hood and within easy reach.
E. For new installations or modifications of existing installations, controls for other services (gas, air, water, etc.) should be located external to the hood and within easy reach.
F. Sash openings should be kept to a minimum.
G. Chemicals and apparatus should be located within the hood should be kept at least 6 inches behind the plane of the sash.
H. Personnel should keep their faces outside the plane of the sash.
I. Storage in hoods should be kept to an absolute minimum.
J. Keep the hazardous materials at least six inches back from the plane of the sash to reduce the amount of chemicals drawn into the operator's breathing zone.

XI. Autoclaves

What do I need to consider when decontaminating my samples using an autoclave?

Autoclaves must be used properly to effectively decontaminate potentially biohazardous materials. The following elements all contribute to autoclave effectiveness.

- Temperature: Adequate chamber temperature is at least 121°C (250°F).
- Time: Adequate autoclaving time is a minimum of 30 minutes, measured after the temperature of the material being sterilized reaches 121°C and 15 psi pressure. The tighter the autoclave is packed, the longer it will take to reach 121°C in the center of the load.
- Contact: Steam saturation of the load is essential for effective decontamination. Air pockets or insufficient steam supply will prevent adequate contact. To ensure adequate steam contact, leave autoclave bags partially open during autoclaving to allow steam to penetrate into the bag. Add a small amount of water inside the bag to help ensure heat transfer to the items being decontaminated (do not add water if it will cause biohazardous materials to splash out of the bag).
- Containers: Use leak-proof containers for items to be autoclaved. Place plastic bags inside a secondary container in the autoclave in case liquids leak out. Plastic or stainless steel containers are appropriate secondary containers. Make sure plastic bags and pans are autoclavable, to avoid having to clean up melted plastic.
- Indicators: Tape indicators can only verify that the autoclave has reached normal operating temperatures for decontamination. Most chemical indicators change color after being exposed to 121°C, but cannot measure the length of time spent at 121°C. Biological indicators (such as Bacillus stearothermophilus spore strips) and certain chemical indicators (such as Sterigage) verify that the autoclave reached adequate temperature for a long enough time to kill microorganisms.
- Use a chemical indicator in every load to monitor the effectiveness of individual autoclave runs (temperature only).
- Once a month, use either a biological indicator (such as Bacillus stearothermophilus spore strips) or a chemical indicator that measures both time and temperature (such as Sterigage). Bury the indicator in the center of the load to validate adequate steam penetration. Keep a log book to record the results.

How do I safely use my autoclave?

Because an autoclave uses saturated steam under high pressure to achieve sterilizing temperatures, proper use is important to ensure operator safety. Prevent injuries when using the autoclave by observing the following guidelines:

- Wear heat resistant gloves, eye protection and a lab coat, especially when unloading the autoclave.
- Prevent steam burns and shattered glassware by making sure that the pressure in the autoclave chamber is near zero before opening the door at the end of a cycle. Slowly open the autoclave door and allow the steam to escape gradually.
- Allow items to cool for 10 minutes before removing them from the autoclave.
- Never put sealed containers in an autoclave. They can explode. Large bottles with narrow necks may also explode if filled too full of liquid.
- Never put solvents, volatile or corrosive chemicals (such as phenol, chloroform, bleach, etc.), or radioactive materials in an autoclave. Call EH&S at 2-5518 if you have questions about proper disposal of these materials.
- Inspect your autoclave components regularly. If you find a problem, notify your area mechanic. Do not operate an autoclave until it has been properly repaired

XII. HYDROFLUORIC ACID

1. Wear neoprene gloves, sleeve covers, apron, foot covering, goggles, and face shield. Do not get in eyes, on skin, or on clothing. **EXERCISE EXTREME CAUTION**.
2. Work only in a fume hood. Do not breathe vapor, mist, or fumes.
3. Do not work alone.
4. Read the hazard alert section of the MSDS for HF.
5. Know the location of emergency shower and eyewash station.
6. Have first aid supplies at hand. (nebulizer and 1% Ca gluconate solution for inhalation burns, 25% Magnesium sulfate and ice or Ca gluconate ointment for skin exposure.)

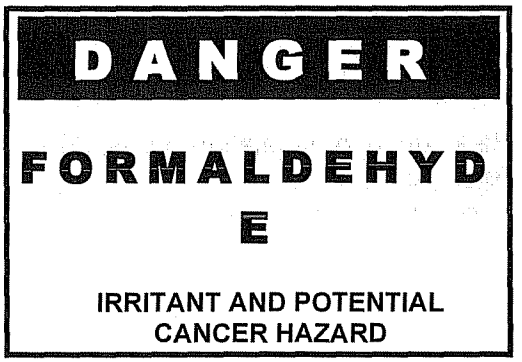
07. If exposed:

SKIN	EYES	INHALED	INGESTED
IMMEDIATELY flush with water. Remove contaminated clothing while in shower (goggles last). Call for emergency medical assistance. If no neutralizer is present continue flushing until medical assistance arrives. Apply 2.5% calcium gluconate gel to skin burns.	IMMEDIATELY flush with copious amounts of water, holding lids open. (Hold eyes open, flush 30 minutes, then instill 1% Ca Gluconate solution). Immediately call for medical assistance.	IMMEDIATELY give oxygen with nebulizer of 1% Ca Gluconate solution. Immediately call for medical assistance.	Do NOT induce vomiting. If victim conscious give 2-4 cups of milk or water. Get medical aid IMMEDIATELY. Speed is of the utmost importance.

Remove all clothing possibly involved. Take victim to hospital after flushing. Take hazard alert info sheet to hospital. Notify emergency room that HF burn case is on the way. If delay, soak burn area (not eyes) with 25% $MgSO_4$ solution (cold). Use ice to reduce pain (no analgesics).

8. CAUTION - Pain can be delayed; even if you suspect that you may have been exposed, treat as above and seek medical treatment.
9. IN CASE OF FIRE, use water spray to cool fire-exposed containers. Use agent appropriate for surrounding fire. Do not get water inside containers. For large fires use spray, fog, or alcohol-resistant foam. DO NOT use straight streams of water. For small fires use carbon dioxide, dry chemical, dry sand, or alcohol-resistant foam.
10. IN CASE OF SPILLS, neutralize spill with sodium bicarbonate. Assure adequate personal protection with clothing and respirator if needed. Use water spray to disperse the gas/vapor. Absorb spill using an absorbent, non-combustible material such as vermiculite or earth. Notify Environmental Health and Safety for assistance with large spills. Avoid runoff into storm drains and ditches.
11. Wash thoroughly after handling.
12. Keep all containers tightly sealed when not immediately in use.
13. Store in a cool, dry, well-ventilated area away from incompatible substances and metals.
14. DO NOT store in metal or glass containers.

XIII. FORMALDEHYDE and FORMALIN
(37% SOLUTION in METHYL ALCOHOL)



Formaldehyde is a colorless, flammable gas with a strong pungent odor. The aqueous solution formalin is 37-40% formaldehyde.

Exposure to formaldehyde vapors can be irritating to the eyes, nose, and upper respiratory tract. In certain individuals, repeated skin exposure to the liquid can cause sensitization that may result in allergic dermatitis.

Formaldehyde is anticipated to be a human carcinogen. Visit www.ehs.ucr.edu for additional information

Minimizing Exposure to Formaldehyde for Health Protection

- Read the MSDS sheet as it pertains to fire hazard, health hazard, exposure treatment, and spill control measures.
- **All work** must be done in a chemical fume hood.
- Wear protective clothing (lab coat, nitrile gloves, safety goggles/ face shield)
- Label all secondary containers with contents and hazard warnings "**TOXIC**" and "**FLAMMABLE**
- Wash thoroughly after handling.
- Know the location of emergency shower and eye wash station and fire extinguisher.

Special Safety Precautions

- If formalin contacts the body/ eyes, flush the affected area with water for at least 15 minutes and report the incident to your supervisor immediately, who will arrange for transport to Riverside Industrial Medical or US Health Works when medical treatment is indicated or requested.

EYES	SKIN	INGESTED	INHALED
Flush with water at least 15 minutes, holding lids open. Call for medical aid immediately.	Flush immediately with soap and water. Remove contaminated clothing. Get medical aid if irritation develops. Wash clothing before reuse, destroy contaminated shoes.	DO NOT INDUCE VOMITING. Only if victim is conscious and alert, give 2-4 cups of milk or water. Call for medical aid immediately.	Call for medical aid immediately. Remove to fresh air. Give artificial respiration if not breathing. Give oxygen if breathing is difficult.

- Contact lenses are best worn in areas where formalin ONLY when chemical splash goggles are used.
- **All solutions** of formalin and tissues preserved in formalin must be stored in tightly sealed containers to prevent leaks, spills, and airborne exposure. Keep away from heat, ignition sources, and strong oxidizers.
- **Don't** pour formalin waste into sinks, place in tightly sealed, labeled waste containers. Visit the EH&S web site to submit waste disposal requests (http://www.ehs.ucr.edu/services/waste.html) or fax request form to 827-5122
- Small spills of diluted formalin solutions must be cleaned up immediately. Cover the spill with paper towels or other absorbent material. **Don't** mop a spill. Using a dust pan, scoop the absorbed formalin into a plastic bag (wear gloves/ eye protection), double bag, seal, and label the waste. Contact EH&S for pickup

Caution If the spill is large, evacuate area immediately; close all doors to contain vapors, and call EH&S during normal business hours, or **9-1-1** after hours. Be prepared to give the location of spill, approximate amount involved, name and phone number. Have someone wait for emergency response personnel outside building and direct them to the spill area.

- **For fires** use dry chemical, carbon dioxide, water spray, or alcohol-resistant foam. Do not use straight streams of water. Collect contaminated water for proper disposal.

XIV. HANDLING Sulfurhexafluoride (SF_6) GAS

GENERAL

- SF6, Sulphur hexafluoride gas, is colourless, odourless, non-flammable and non-toxic (if not heated above 250 °C).
-The gas is heavier than air.
- The gas causes suffocation at high concentration levels.
- The gas breaks down when overheated 2' giving off highly toxic fluorine gas.

THEREFORE:

- The ventilation system must be switched to extract AT ALL TIMES.
- Smoking in SF_6 sensitive areas is FORBIDDEN.
- The SF6 detector must ALWAYS be in operation.
- Gas masks (2x) and rubber gloves (2 pairs) MUST BE PRESENT.
    - For transporting the gas, use the tools and equipment delivered
    - For complete safety, an SF6 detector* should be present and operating CONTINUOUSLY. The detector must be a suitably calibrated instrument using the thermal conductivity or infrared process principle.

WHAT TO DO IN EVENT OF A GAS LEAKAGE - Switch off all the heat sources – MAKE SURE the VENTILATION SYSTEM IS VORKING AND CLOSE THE DOOR. Trace and stop the leakage.
SF6 gas is heavier than air, thus it will sink and stay at floor level. There should therefore be an extraction ventilator channel opening about 10-15 cm above the floor. This ventilation channel should open directly into the outside air and must UNDER NO CIRCUMSTANCES BE CONNECTED TO THE CENTRAL VENTILATION SYSTEM OF THE BUILDING. CAPACITY 100 M^3 /HR.
All possible holes in the floor giving access to lower floors in the building should be closed.

When a filament (wehnelt) of the CM200 is replaced, a certain amount of SF_6 gas must be pumped away with the p.v. pump of the microscope. Because of this, the outlet of p.v. pump should be connected to the atmosphere outside the building and NEVER UNDER ANY CIRCUMSTANCES to the central ventilation system of the building.
The safety blow-off valve of the H.T. tank must also be connected to the SF_6 ventilation system in order to extract SF6 gas in the event of an overpressure failure.

WARNING! In the event of fire, SF_6 will decompose to highly poisonous fluorine if the temperature exceeds 250°C.

WHAT TO DO IF SF6 DECOMPOSES AND FLUORINE IS GIVEN OFF
Note: Even the presence of very low concentrations of fluorine can be detected by smell.

- Put on a gas mask with filters of active charcoal and incorporated dust filter.
- Ventilate the area (but DO NOT OPEN THE DOOR).
- Switch off heat sources.

- Trace and stop the leakage
- All parts contaminated with solid decomposed products must be handled with rubber gloves.

SPECIFICATIONS

The SF6 gas used must meet the following specifications:

SF6 (IEC)	Esaflon			
SF6 minimum contents		Weight %	99.9	99.94
Air	Weight %	max. 0.05	max. 0.01	
CF4	Weight %	max. 0.05	max. 0.05	
Water	Weight ppm	max. 15	max. 2	
Acidity, calculated as HF		Weight ppm	max. 0.3	max. 0.3
Hydrolysable fluoride, calculated as HF		Weight ppm	max. 1	max. 1
Mineral oil		Weight ppm	max. 10	max. 1

The MONTEDISON company is a manufacturer of SF_6 gas.

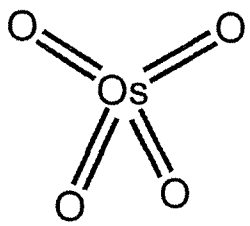
XV. HANDLING *Osmium Tetroxide* (OsO_4)

General

Osmium tetroxide is a volatile, highly toxic solid that is used as a stain for electron microscopy, and as a fixative for biological samples.

Physical & Chemical Properties/Definition of Chemical Group

Synonyms: Osmium tetroxide
Osmic acid
Perosmic oxide
Osmium(IV) oxide



CAS#: 20816-12-0

Molecular formula: OsO_4

Molecular weight: 254.23 g

Form: pale yellow solid

Potential Hazards/Toxicity

Toxic Effects: Oxidizing material. Highly toxic. Contact with combustible materials may cause fire. May cause sensitization by inhalation or skin contact.

Acute Effects: The acute toxicity of osmium tetroxide is high, and it is a severe irritant of the eyes and respiratory tract.

Exposure to osmium tetroxide vapor can damage the cornea of the eye. Irritation is generally the initial symptom of exposure to low concentrations of osmium tetroxide vapor, and lacrimation, a gritty feeling in the eyes, the eyes can temporarily cloud, and the appearance of rings around lights may also be noted. In most cases, recovery occurs in a few days. Concentrations of vapor that do not cause immediate irritation can have an insidious cumulative effect; symptoms may not be noted until several hours after exposure. Contact of the eyes with concentrated solutions of this substance can cause severe damage and possible blindness.

Inhalation can cause headache, coughing, dizziness, lung damage, difficult breathing and may be fatal.

Contact of the vapor with skin can cause dermatitis, and direct contact with the solid can lead to severe irritation and burns. Exposure to osmium tetroxide via inhalation, skin contact, or ingestion can lead to systemic toxic effects involving liver and kidney damage. Osmium tetroxide is regarded as a substance with poor warning properties.

Chronic Effects: Chronic phenol poisoning is characterized by vomiting, difficult swallowing, excessive salivation, diarrhea, anorexia, headache, fainting, vertigo, mental disturbances, and possibly skin eruptions. Prolonged cutaneous exposure may result in deposition of dark pigment in the skin.

Exposure Limits: Cal-OSHA PEL= 0.0002 ppm (0.002 mg/m^3)

Personal Protective Equipment (PPE)

- Chemical goggles (safety glasses alone are not adequate protection because of osmium tetroxide's severe effects on the eyes).
- Disposable nitrile gloves (NOT latex). Double-gloving is recommended when working with pure osmium tetroxide or concentrated solutions. Change gloves frequently and when contaminated, punctured or torn. Wash hands immediately after removing gloves.
- A standard or disposable laboratory coat or disposable coveralls. A standard laboratory coat may be reused before laundering if it has not been contaminated with osmium tetroxide. If a garment is contaminated, remove, place in chemical hood, and decontaminate with corn oil or aqueous solutions of sodium sulfide or sodium sulfite before disposing of in hazardous waste or laundering.
- Long pants and closed-toed shoes must be worn.
- Wash hands thoroughly immediately after working with any concentration of osmium tetroxide.

Controls

- Osmium tetroxide solutions must be prepared and handled in a certified chemical hood.
- Choose a hood with minimal equipment or obstructions to ensure good containment of vapors.
- Working surfaces should be protected with plastic backed absorbent pads to insure containment of any spills.
- Post the hood with a warning sign to alert others to the hazards, see warning sign at the end of this SOP.
- Ensure that the safety shower and eyewash are operational and access is unblocked.

First Aid Procedures

Eye Contact: Check for and remove any contact lenses. Immediately flush eyes with running water for at least 15 minutes, keeping eyelids open. Cold water may be used. Do not use an eye ointment. Seek medical attention.

Skin Contact: After contact with skin, wash immediately with plenty of water. Gently and thoroughly wash the contaminated skin with running water and non-abrasive soap. Be particularly careful to clean folds, crevices, creases and groin. Cold water may be used. Cover the irritated skin with an emollient. If irritation persists, seek medical attention.

Serious Skin Contact: Wash with a disinfectant soap and cover the contaminated skin with an anti-bacterial cream. Seek immediate medical attention.

Inhalation: Allow the victim to rest in a well ventilated area. Seek immediate medical attention.

Serious Inhalation: Evacuate the victim to a safe area as soon as possible. Loosen tight clothing such as a collar, tie, belt or waistband. If breathing is difficult, administer oxygen. If the victim is not breathing, perform mouth-to-mouth resuscitation. Seek medical attention.

Ingestion: Do not induce vomiting. Examine the lips and mouth to ascertain whether the tissues are damaged, a possible indication that the toxic material was ingested; the absence of such signs, however, is not conclusive. Loosen tight clothing such as a collar, tie, belt or waistband. If the victim is not breathing, perform mouth-to-mouth resuscitation. Seek immediate medical attention.

Special Handling and Storage Requirements

Precautions: Keep away from heat. Keep away from sources of ignition. Keep away from combustible material. Do not ingest. Do not breathe dust. Wear suitable protective clothing In case of insufficient ventilation, wear suitable respiratory equipment If ingested, seek medical advice immediately and show the container or the label. Avoid contact with skin and eyes.

Storage: Keep container tightly closed. Keep in a cool, well-ventilated place. Highly toxic or infectious materials should be stored in a separate locked safety storage cabinet or room.

Because of osmium tetroxide's high acute toxicity and powerful oxidizing ability, osmium tetroxide must be handled in the laboratory using prudent practices. In particular, all work with osmium tetroxide must

be conducted in a fume hood to prevent exposure by inhalation, and personal protective equipment (see section) must be worn at all times to prevent eye and skin contact.

When osmium tetroxide is freshly prepared and active, it is colorless to pale yellow in color. When the material reacts and causes oxidation, it turns black. This is helpful to know especially in the event of a splash or spill (see below) or inadvertent dermal exposure (black dots on skin).

- Pure osmium tetroxide and concentrated solutions should be stored in a location that is secure to unauthorized access.
- A refrigerator containing osmium tetroxide must be labeled with a caution sign noting the presence of osmium tetroxide and its hazards.
- Store pure osmium tetroxide and its concentrated solutions in appropriate, sealed glass containers within unbreakable secondary containment (i.e., a bottle or vial within a sealed compatible plastic jar or metal can with lid). Label all containers, including secondary containment, with the chemical name and hazard warning.

Handling and Solution Preparation:

- When moving pure osmium tetroxide to a chemical hood, do not remove it from the secondary containment until it is in the hood.
- Prepare the smallest amount of solution necessary for the procedure, typically 50 mL or less. Prepare the solution volumetrically rather than gravimetrically. If a balance must be used, weighing must take place in the chemical hood.
- Pure osmium tetroxide or its concentrated solutions must be opened only in a chemical hood that has been certified within the last 12 months. Just before use, the operation of the chemical hood must be verified by the use of an installed chemical hood monitoring device, a smoke test using a smoke generating tube, or a mechanical or electronic device that indicates air flow. During use, the sash must be lowered to operating height.
- All lab ware that has contacted osmium tetroxide must be decontaminated by rinsing or dipping in corn oil or aqueous solutions of sodium sulfide or sodium sulfite before removing from the hood.
- Immediately after work with osmium tetroxide, decontaminate any spills with kitty litter soaked with corn oil. Discard kitty litter as hazardous waste. Or use aqueous solutions of sodium sulfide or sodium sulfite.

Spill and Accident Procedure

Chemical Spill Dial 911 and x2522

Spill – Help contaminated or injured persons. Evacuate the spill area. Avoid breathing vapors. Eliminate sources of ignition if the chemical is flammable. If possible, confine the spill to a small area using a spill kit or absorbent material. Keep others from entering contaminated area (e.g., use caution tape, barriers, etc.).

Small (<1 L) – If you have training, you may assist in the clean-up effort. Use appropriate personal protective equipment and clean-up material for chemical spilled. Double bag spill waste in clear plastic bags, label and take to the next chemical waste pick-up.

Large (>1 L) – Dial **911** and EH&S at x2522 for assistance.

Chemical Spill on Body or Clothes – Remove clothing and rinse body thoroughly in emergency shower for at least 15 minutes. Seek medical attention. *Notify supervisor and EH&S at x2522 immediately.*

Chemical Splash Into Eyes – Immediately rinse eyeball and inner surface of eyelid with water for 15 minutes by forcibly holding the eye open. Seek medical attention. *Notify supervisor and EH&S at x25222 immediately.*

Medical Emergency Dial 911 or x25222

Decontamination/Waste Disposal Procedure

- To reduce hazards involved in discarding osmium tetroxide, the following neutralization procedure should be employed:

1. Perform neutralization in a chemical hood.
2. A 2% solution of osmium tetroxide can be fully neutralized by twice its volume of vegetable oil (corn oil is preferred because of its high percentage of unsaturated bonds). For every 10 mL of 2% osmium textroxide solution, 20 mL of corn oil is required. Pour the corn oil into the osmium tetroxide solution.
3. Wait for the oil to completely turn black.
4. To test if osmium tetroxide is fully neutralized, hold a piece of filter paper soaked in corn oil over the solution. Blackening indicates that osmium tetroxide is still present and more corn oil should be added.
5. Aqueous solutions contaminated with osmium tetroxide can be fully neutralized by adding sodium sulfide or sodium sulfite to reduce osmium tetroxide to less hazardous forms.
6. Dispose of neutralized solutions as hazardous waste.

- To dispose of waste:

SOLUTIONS: DO NOT dispose of leftover or spent solutions of osmium tetroxide by emptying into the laboratory sink. Collect solutions and solid osmium tetroxide in a labeled leak proof waste container for pickup and proper disposal by EH&S Hazardous Waste Pickup service. A completed waste label must be attached to the container.

SOLIDS: Collect pipette tips, gloves, ampoules, etc in a rigid leak proof container. Affix a completed waste label to the container. Due to the toxicity of osmium tetroxide, empty containers must also be collected and disposed as hazardous waste.

Appendix B – SAFETY & HAZARD Information Sources at UCR

A: *Prudent Practices for Handling Hazardous Chemicals in Laboratories*, 1981, National Research Council, Washington D.C.: National Academy Press. UCR Science Library QD51 N32x 1981

B: *CRC Handbook of Laboratory Safety*, 5th ed., 2000, A.K. Furr, ed., Boca Raton, Fla.: CRC Press. UCR Science Library Ref QD51 H27 2000.

C: *Improving Safety in the Chemical Laboratory: A Practical Guide*, 2nd ed., 1991, J. A. Young, ed., New York:Wiley. UCR Science Library QD51 I48 1991

D: *Safe Storage of Laboratory Chemicals*, 1984, A. Pipetone, ed., New York:Wiley. UCR Science Library QD51 S22 1984 (1991 ed. in EH&S Office)

E: *Chemical Compatibility/Segregation*, from the EH&S website, http://www.ehs.ucr.edu/resources/wastecategory.pdf

F: *Hazardous Chemicals Desk Reference*, 4th ed., 1997, R.J. Lewis, Sr., ed., New York:Van Nostrand Reinhold. UCR Science Library Ref T55.3.H3 L49 1997.

G: *Patty's Industrial Hygiene and Toxicology*, 4th ed., 1991, G. Clayton and F.E. Clayton, eds., New York:Wiley. UCR Science Library Ref RC967 P37 1991

H: *Rapid Guide to Chemical Incompatibilities*, 1997, R.P. Pohanish and S. A. Greene, New York:Van Nostrand Reinhold. UCR Science Library Ref T55.3 H3 P644 1997

I: *Toxic and Hazardous Industrial Chemicals Safety Manual*, 1988, The International Technical Information Institute, Tokyo:Japan. UCR Science Library Ref T55.3 H3 K34 1988

J: *Handbook of Laboratory Health and Safety*, 2nd ed., 1995, R.S. Stricoff and D.B. Walters, New York:Wiley. UCR Science Library QD51 S92 1995

K. *Dangerous Properties of Industrial Materials*, 7th ed., 1987, N.I. Sax and R.J. Lewis Sr., (eds). New York, Van Nostrand Reinhold. UCR Science Library T55.3.H3 S3 1988 Ref.

L. *NIOSH Pocket Guide to chemical Hazards*, 1990, U.S. Dept. of Health and Human Services, Public Health Service, CDC, NIOSH. June, 1990.

M. UCR Environmental Health and Safety (EH&S) office (951)827-5528, web site: http://www.ehs.ucr.edu/ and for Material Safety Data Sheet and Chemical Safety Information: http://www.ehs.ucr.edu/services/msds.html

Appendix C Health hazard definitions and hazard assessment

[Appendix A to CCR Title 8 section 5194 Health Hazard Definitions, http://www.dir.ca.gov/Title8/5194a.html]

Although safety hazards related to the physical characteristics of a substance can be objectively defined in terms of testing requirements (e.g., flammability), health hazard definitions are less precise and more subjective. Health hazards may cause measurable changes in the body - such as decreased pulmonary function. These changes are generally indicated by the occurrence of signs and symptoms in the exposed employees such as shortness of breath, a non-measurable, subjective feeling. Employees exposed to such hazards must be apprised of both the changes in body function and the signs and symptoms that may occur to signal that change.

The determination of occupational health hazards is complicated by the fact that many of the effects or signs and symptoms occur commonly in non-occupationally exposed populations, so that effects of exposure are difficult to separate from normally occurring illnesses. Occasionally, a substance causes an effect that is rarely seen in the population at large, such as angiosarcomas caused by vinyl chloride exposure, thus making it easier to ascertain that the occupational exposure was the primary causative factor. More often, however, the effects are common, such as lung cancer. The situation is further complicated by the fact that most substances have not been adequately tested to determine their health hazard potential, and data do not exist to substantiate these effects.

There have been many attempts to categorize effects and to define them in various ways. Generally, the terms "acute" and "chronic" are used to delineate between effects on the basis of severity or duration. "Acute" effects usually occur rapidly as a result of short-term exposures, and are of short duration. "Chronic" effects generally occur as a result of long-term exposure, and are of long duration.

The acute effects referred to most frequently are those defined by the American National Standards Institute (ANSI) standard for Precautionary Labeling of Hazardous Industrial Chemicals (Z129.1- 1982) - irritation, corrosivity, sensitization and lethal dose. Although these are important health effects, they do not adequately cover the considerable range of acute effects that may occur as a result of occupational exposure, such as, for example, narcosis.

Similarly, the term chronic effect is often used to cover only carcinogenicity, teratogencity, and mutagenicity. These effects are obviously a concern in the workplace, but again, do not adequately cover the considerable range of chronic effects, excluding, for example, blood dyscrasia (such as anemia), chronic bronchitis and liver atrophy.

The goal of defining precisely, in measurable terms, every possible health effect that may occur in the workplace as a result of substance exposures cannot realistically be accomplished. This does not negate the need for employees to be informed of such effects and protected from them.

At http://www.dir.ca.gov/Title8/5194b.html Appendix B of the California Code of Regulations outlines the principles and procedures of hazard assessment.

For purposes of this section, any substances that meet any of the following definitions, as determined by the criteria set forth in that Appendix B are health hazards:

1. **Carcinogen**: A substance is considered to be a carcinogen if:

a. It has been evaluated by the International Agency for Research on Cancer (IARC) Monographs, Vols 1-34, and found to be a carcinogen or potential carcinogen; or

b. It is listed as a carcinogen or potential carcinogen in the Third Annual Report on Carcinogens published by the National Toxicology Program (NTP); or

c. It is regulated by OSHA as a carcinogen.

2. **Corrosive**: A substance that causes visible destruction of, or irreversible alterations in, living tissue by chemical action at the site of contact. For example, a substance is considered to be corrosive if, when tested on the intact skin of albino rabbits by the method described by the U.S. Department of Transportation in Appendix A to 49 CFR Part 173, it destroys or changes irreversibly the structure of the tissue at the site of contact following an exposure period of four hours. This term shall not refer to action on inanimate surfaces.

3. **Highly Toxic**: A substance falling within any of the following categories:

a. A substance that has a median lethal dose (LD50) of 50 milligrams or less per kilogram of body weight when administered orally to albino rats weighing between 200 and 300 grams each.

b. A substance that has a median lethal dose (LD50) of 200 milligrams or less per kilogram of body weight when administered by continuous contact for 24 hours (or less if death occurs within 24 hours) with the bare skin of albino rabbits weighing between two and three kilograms each.

c. A substance that has a median lethal concentration (LC50) in air of 200 parts per million by volume or less of gas or vapor, or 2 milligrams per liter or less of mist, fume, or dust, when administered by continuous inhalation for one hour (or less if death occurs within one hour) to albino rats weighing between 200 and 300 grams each.

4. Irritant: A substance that is not corrosive but that causes a reversible inflammatory effect on living tissue by chemical action at the site of contact. A substance is a skin irritant if, when tested on the intact skin of albino rabbits by the methods of 16 CFR 1500.41 for four hours exposure or by other appropriate techniques, it results in an empirical score of five or more. A substance is an eye irritant if so determined under the procedure listed in 16 CFR 1500.42 or other appropriate techniques.

5. **Sensitizer**: A substance that causes a substantial proportion of exposed people or animals to develop an allergic reaction in normal tissue after repeated exposure to the substance.

6. **Toxic**: A substance falling within any of the following categories:

a. A substance that has a median lethal dose (LD50) of more than 50 milligrams per kilogram but not more that 500 milligrams per kilogram of body weight when administered orally to albino rats weighing between 200 and 300 grams each.

b. A substance that has a median lethal dose (LD50) of more than 200 milligrams per kilogram but not more than 1,000 milligrams per kilogram of body weight when administered by continuous contact for 24 hours (or less if death occurs within 24 hours) with the bare skin of albino rabbits weighing between two and three kilograms each.

c. A substance that has a median lethal concentration (LC50) in air of more than 200 parts per million but not more than 2,000 parts per million by volume of gas or vapor, or more than two

milligrams per liter but not more than 20 milligrams per liter of mist, fume, or dust, when administered by continuous inhalation for one hour (or less if death occurs within one hour) to albino rats weighing between 200 and 300 grams each.

7. **Target Organ Effects**: The following is a target organ categorization of effects that may occur, including examples of signs and symptoms and substances that have been found to cause such effects. These examples are presented to illustrate the range and diversity of effects and hazards found in the workplace, and the broad scope employers must consider in this area, but are not intended to be all-inclusive.

   a. Hepatotoxins: Substances that produce liver damage.
   Signs and Symptoms: Jaundice, liver enlargement.
   Substances: Carbon tetrachloride; nitrosamines.

   b. Nephrotoxins: Substances that produce kidney damage.
   Signs and Symptoms: Edema, proteinuria.
   Substances: Halogenated hydrocarbons, uranium.

   c. Neurotoxins: Substances that produce their primary toxic effects on the nervous system.
   Signs and Symptoms: Narcosis, behavioral changes, decreases in motor functions.
   Substances: Mercury, carbon disulfide.

   d. Agents that act on the blood or hematopoietic system, decrease hemoglobin function, deprive the body tissues of oxygen.
   Signs and Symptoms: Cyanosis, loss of consciousness.
   Substances: Carbon monoxide, cyanides.

   e. Agents that damage the lung, substances which irritate or damage the pulmonary tissue.
   Signs and Symptoms: Cough, tightness in chest, shortness of breath.
   Substances: Silica, asbestos.

   f. Reproductive toxins: Substances that affect the reproductive capabilities including chromosomal damage (mutations) and effects on fetuses (teratogenesis).
   Signs and Symptoms: Birth defects, sterility.
   Substances: Lead, DBCP.

   g. Cutaneous hazards: Substances that affect the dermal layer of the body.
   Signs and Symptoms: Defatting of the skin, rashes, irritation.
   Substances: Ketones, chlorinated compounds.

   h. Eye hazards: Substances that affect the eye or visual capacity.
   Signs and Symptoms: Conjunctivitis, corneal damage.
   Substances: Organic solvents, acids.

Hazard Determination (http://www.dir.ca.gov/Title8/5194b.html)

The quality of a hazard communication program is largely dependent upon the adequacy and accuracy of the hazard determination. The hazard determination requirement of this standard is performance-oriented. Manufacturers, importers, and employers evaluating substances are not required to follow any specific methods for determining hazards, but they must be able to demonstrate that they have adequately ascertained the hazards of the substances produced or imported in accordance with the criteria set forth in this Appendix.

Hazard evaluation is a process that relies heavily on the professional judgment of the evaluator, particularly in the area of chronic hazards. The performance orientation of the hazard determination does not diminish the duty of the manufacturer, importer or employer to conduct a thorough evaluation, examining all relevant data and producing a scientifically defensible evaluation. For purposes of this standard, the following criteria shall be used in making hazard determinations that meet the requirements of this standard.

1. Carcinogenicity:

As described in subsection 5194 (d)(4) and (http://www.dir.ca.gov/Title8/5194a.html), a determination by the National Toxicology Program, the International Agency for Research on Cancer, or OSHA that a substance is a carcinogen or potential carcinogen will be considered conclusive evidence for purposes of this section.

2. Human Data:

Where available, epidemiological studies and case reports of adverse health effects shall be considered in the evaluation.

3. Animal Data:

Human evidence of health effects in exposed populations is generally not available for the majority of substances produced or used in the workplace. Therefore, the available results of toxicological testing in animal populations shall be used to predict the health effects that may be experienced by exposed workers. In particular, the definitions of certain acute hazards refer to specific animal testing results (see Appendix A http://www.dir.ca.gov/Title8/5194a.html).

4. Adequacy and Reporting of Data:

The results of any studies that are designed and conducted according to established scientific principles, and which report statistically significant conclusions regarding the health effects of a substance, shall be a sufficient basis for a hazard determination and reported on any material safety data sheet. The manufacturer, importer, or employer may also report the results of other scientifically valid studies that tend to refute the findings of hazard.

Appendix D

Sample Posting for Designated Areas

WARNING:
Designated Area
for use of
*****

*** Insert one or more as appropriate - "Select Carcinogens", "Reproductive Toxins", "Highly Toxic Chemicals"**

Appendix E LAB SAFETY & ENVIRONMENTAL ASSESSMENT AUDIT

GENERAL HOUSEKEEPING	Y	N	*
1 **Hallways clear** of chemicals, combustibles, surplus equipment and debris for the required exit width. For a fire rated corridor this is 44" min. If the space allows, any materials in the hall must be **secured** along one side, so as to prevent the creation of a convoluted path.			
2 **Aisles** in the lab clear and wide, 36 in or more whenever possible.			
3 **Bench tops** clear, organized and maintained to eliminate harmful exposures/unsafe conditions. **Food and drink not consumed or stored** in chemical use areas or refrigerators.			
4 **Fume hood** chemical storage is minimized and not blocking airflow to slots in the baffle. Use is generally six inches from front edge and sash below 18" or certification mark.			
5 **Shelves** are relatively clean &, well organized.			
6 **Storage cabinets** are clean & well organized. Within one room, in excess of 10 gal of flammable liquids are stored in approved flammable storage cabinets.			
GENERAL LABORATORY SIGNS			
7 Are **eyewash/safety shower** visible within service area or signs are posted to location?			
8 Are disaster & **emergency** response lists **posted and visible**?			
9 Are the **doors** labeled with up to date, **responsible party** & hazards signs? (check the placard)			
10 Are **OSHA safety & health protection on the job** & **In case of injury** posters in a common area?			
11 Are "**Danger: High Voltage**" signs posted when appropriate (electrophoresis, etc.)?			
12 Are "**lasers are in use**" caution signs posted (where appropriate)?			

HAZARDOUS MATERIALS STORAGE AREA SIGNS

13 Do all flammable storage locations have **"FLAMMABLES KEEP FIRE AWAY** signs posted?

14 Do all hazardous material storage locations have the appropriate **hazard warning signs** posted?
(❍ Corrosive (☐ acid & ☐ base); ❍ Oxidizer; ❍ Toxic; ❍ Highly Toxic or Poisons; ❍ Biohazard;
❍ Radioactive)

15 When Cal/OSHA regulated **Carcinogens** are stored and/or used, is the designated use area posted with the notice of use? (commonly used are: methylene chloride (a.k.a. dichloromethane, benzene & formaldehyde for complete list: www.dir.ca.gov/title8v/sb7g16a110.html)

16 Are **radiation signs** and labels posted where **radioactive materials** are used and/or stored?

17 Are **biohazards** signs and labels posted where **biohazardous materials** are used and/or stored?

18 Are **all containers labeled with complete chemical names** and associated **hazards** or an **abbreviation system** posted with this information in every room where these abbreviations are used on containers?

SEISMIC ISSUES

19 Are all **Cabinets**, shelf units (> 42") and equipment seismically **restrained?**

20 Do all **shelves** have **seismic lips** or other restraints?

21 Are **flexible** hose **connections** used for all lab equipment and apparatus to connect to utilities?

CHEMICAL STORAGE

22 **Are all chemicals segregated by hazard class?**
Circle these classes that stored together:
a. separate **oxidizers** from *organics*, *reducing reagents* and *combustibles*;
b. separate powerful **reducing agents** from *readily reduced substrates*;
c. **pyrophoric** compounds from *flammables*;
d. **acids** from *bases*,

23	Is the use of **secondary containment** for inventory common? (Corrosion resistant storage trays or tubs that can hold 110 % of the volume of the largest liquid container. For solids, select a convenient size.)			
24	All acids and caustics stored **below eye level** wherever practical?			
25	All **pesticide storage** cabinets/areas are labeled as such, locked with MSDS readily available?			
26	**Controlled substances** in locked location? (DEA regulated materials)			
27	Are all compressed **gas** cylinders **segregated** into hazard classes and stored separate from other chemicals. Empty cylinders clearly marked.			
28	Are all **compressed** gas **cylinders** stored upright, **secured** from falling and checked for both current use and functioning valves at least annually?			
29	**ARE** ALL FLAMMABLE MATERIALS **STORED** AWAY **FROM** IGNITION **SOURCES?**			
30	NO FLAMMABLE LIQUIDS **ARE BOTTOM DISPENSED FROM** NON-FLASH ARRESTOR, NON-SELF-CLOSING **BOTTOM, G**RAVITY DISPENSING **CONTAINER, RIGHT?**			
31	All **chemical storage cabinets are vented** to the fume hood duct exhaust?			
32	All **time sensitive materials** (such as peroxide formers) are marked with the date when they arrive, when they are opened and are not retained past the expiration date?			
ELECTRICAL EQUIPMENT				
40	Only **grounded or double-insulated** electrical equipment is used?			
41	**Extension cords** are all used on a temporary basis only (< 8hr), except power strips?			
42	There are no **trip-hazards** in walkways or **frayed cords** (exposed live current wires), right?			
HAZARDOUS WASTE				
44	All chemical, biological and radioactive hazardous wastes located and managed to **prevent cross-contamination**?			
45	All **liquids wastes** are kept in **secondary containers**, gray tubs?			

46	All hazardous wastes are kept in containers with **positive closures** (screw-top)? (no beakers, etc)			
47	All hazardous wastes containers are **kept closed** except when actively adding waste?			
48	All **chemical wastes** are **segregated** by hazard class, right?			
49	All **chemical wastes** are **properly labeled** as waste with chemical names and type of hazard and satellite accumulation area start dates using the UCR hazardous waste labels?			
50	No waste is accumulated for longer than **6 months**, right?			
51	**All biohazardous** waste is properly labeled and disposed, right? • All bags labeled with room and date and stored in a rigid, leadkproof, container with tight fitting lid. • No bags stored for 1 week or more.			
52	All biohazards and medical waste is collected in **RED bags** and containers, right?			
53	All **radioactive** wastes are segregated, labeled with isotope and radiation sign? • If chemicals are in the same container the hazardous waste label is posted as well, right?			
54	All **sharps waste** is properly disposed in rigid containers that do not allow for sharp edges or points to protrude, right?			

ENGINEERING CONTROLS

55	**Room ventilation** vents are clear of obstructions, air quality is adequate, the pressure in labs or rooms with hazardous materials is negative to the halls and offices, right?			
56	All **fume hoods** are **certified** within last year and a functioning air flow indicator is present, yes?			
57	**Biological safety cabinets - certified** within last year.			

PERSONAL PROTECTIVE EQUIPMENT

58	Is **safety eyewear** (safety glasses, goggles, laser goggles, face shield, etc.) available, stored properly & **worn** when in the lab?			
59	Are **gloves** appropriate to the task available & reusable gloves inspected before each use, properly stored?			
60	**Lab coats & aprons** available & worn when needed.			

61	**Respiratory protection** available when engineering controls cannot be used, stored properly, and worn when needed.			
62	All appropriate personal protective equipment available, worn when needed and **open-toed footwear is forbidden** in the lab.			

EMERGENCY EQUIPMENT & SUPPLIES

63	**Safety shower/eyewash located** within 10 seconds travel from hazardous materials use areas and the access is kept clear.			
64	**Safety shower/eyewash tested** and recorded **monthly** by physical plant			
65	**Fire extinguishers** easy to access, and inspected monthly, or at more frequent intervals, and serviced at least annually. CCR 8, §1922(a)(4)			
66	**Circuit breaker panel** at least 30 in. clear access, circuits labeled.			
67	**Spill kits** present, appropriate e.g., the use of hydrofluoric acid requires special material), and kept well supplied.			
68	**First aid kits** present & well supplied. Materials with limited shelf-life should be removed when expired. (No ammonia inhalant ampoules.)			
69	**Telephone** access for emergencies within the area at all hours.			

DOCUMENTS that are required by law to be available to lab personnel

The Departmental Injury & Illness Prevention Program
The Departmental Chemical Hygiene Plan
- The written procedures for using chemical safely (Standard Operating Procedures SOPs)

Records of safety training provided in the department (with the employees signature)
Exposure control plan for bloodborne pathogens has been completed if using, blood, blood products, human tissue or human cells or cell lines.

Appendix F

Inspection Log

Protective Goggles		Cryogenic Gloves		Protective Aprons	
Initials	Date	Initials	Date	Initials	Date